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THE IDENTIFICATION OF FLAX, HEMP, JUTE AND RAMIE IN TEXTILE ARTIFACTS

BY

JOAN A. MARSHALL

©

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND RESEARCH IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

MASTER OF SCIENCE

DEPARTMENT OF CLOTHING AND TEXTILES

EDMONTON, ALBERTA

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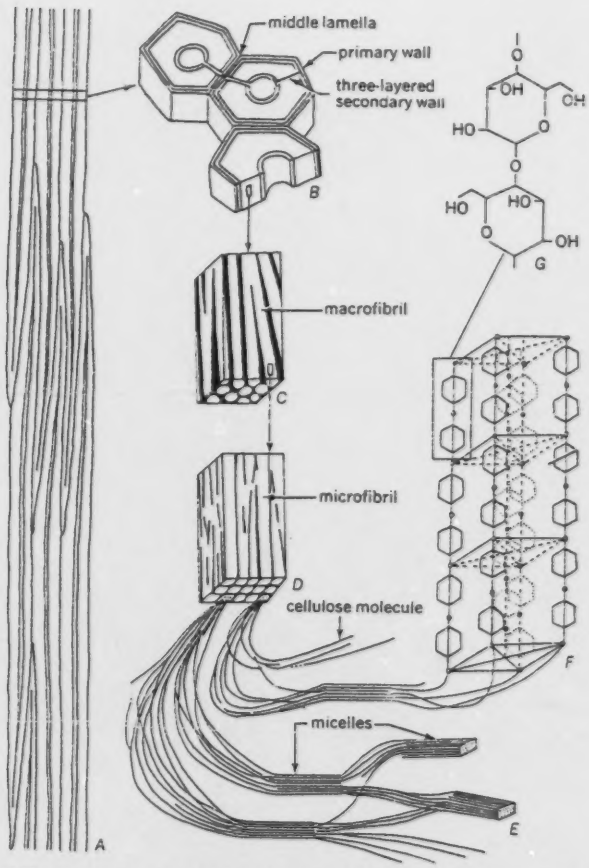

Detailed structure of cell walls. *A*, strand of fiber cells. *B*, cross section of fiber cells showing gross layering: a layer of primary wall and three layers of secondary wall. *C*, fragment from middle layer of secondary wall showing macrofibrils (white) of cellulose and interfibrillar spaces (black), which are filled with noncellulosic materials. *D*, fragment of a macrofibril showing microfibrils (white), which may be seen in electron micrographs (fig. 4.2). The spaces among microfibrils (black) are filled with noncellulosic materials. *E*, structure of microfibrils: chainlike molecules of cellulose, which in some parts of microfibrils are orderly arranged. These parts are the micelles. *F*, fragment of a micelle showing parts of chainlike cellulose molecules arranged in a space lattice. *G*, two glucose residues connected by an oxygen atom—a fragment of a cellulose molecule.

Figure 2

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Dr.N. Kerr (supervisor)

Dr. E. Crown

Dr. D. Cass

Date: September 22, 1992

ABSTRACT

The identification of bast fibres in textile artifacts is difficult to accomplish. Methods developed have been used with varying results among researchers. For a method to be successful, it must either highlight unique structural details or cause unique behaviour in a fibre. When fibres are degraded, the detection of any features that persist must also be facilitated.

This research initially set out to develop a scheme that could make possible the identification of flax, hemp, jute and ramie. Methods selected for this research showed consistent and precise results during trial testing. The second objective was to determine whether there are characteristics that persist as these fibre species are degraded and if such characteristics are useful in identifying unknown fibres. The third objective was to attempt to determine the identification of twenty unknown hard or soft vegetable fibres using the identification scheme developed with known specimens.

First, specimens of flax, hemp, jute and ramie were tested and observed. Three chemical tests were done using phloroglucinol and hydrochloric acid, Herzberg reagent and Schweitzer's reagent. Then, the twist test was administered, fibre diameters were measured and observations by optical and scanning electron microscope were made.

Results of the tests and observations were remarkably consistent within each species in the known specimens. Measurements also showed some consistency, though ranges and standard deviations were sometimes quite wide.

The group of tests administered to the known specimens was then administered to twenty unknown specimens. The less degraded a specimen was, the more conclusive was the identification that could be made. Though some identifications were tentative, most of the unknowns were identified as flax, with three as hemp, one as jute, and three remaining unidentified.

It was concluded that careful control of test methods is important for obtaining consistent results from specimens. Administering the entire testing scheme to all unknown fibres, no matter how degraded, is important as it can not necessarily be known to which tests a fibre will or will not respond. When a fibre shows little response or gives confusing results, provenance is important in helping to identify that fibre species.

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CHAPTER 1 INTRODUCTION

The Franklin Expedition Textile Artifacts

Between 1846 and 1848, 129 men from the third Sir John Franklin British Arctic Expedition perished seeking the Northwest Passage to the Pacific Ocean. Speculation about what happened to these people and why none of them survived the journey has existed from that time until 1987. In 1986, the body of John Torrington was exhumed by Dr. Owen Beattie and his team. The following year, the bodies of John Hartnell and William Braine were also retrieved. Before reburial, tissue and textile samples were taken from them (Kerr & Schweger, 1989).

The members of the Beattie team, all part of the Franklin Forensic Project, also traced the known route as recorded by Franklin as well as what they interpreted to be the subsequent route (Beattie & Geiger, 1988). Throughout the journey interesting items were found on the ground. Though far more weathered and degraded than the grave artifacts, recognizable pieces of such things as food cans, shoe leather, glass and pieces of rope, cordage and canvas were collected. The rope and canvas pieces, though possibly from slightly later expeditions, often had lichen and moss growing on them. They also showed evidence of other forms of biological deterioration. Some of the pieces still carried the residue of resins or some such material with which they had been impregnated as a waterproofing agent.

The book, *Frozen in Time*, (Beattie & Geiger, 1988), refers to the finding of some of the cordage artifacts:

> Visible in the mud were coils of rope of various sizes. The anaerobic conditions of the mud, and the long period of freezing each year, had resulted in good preservation of the organic material, in stark contrast to the disintegrated rope and canvas fragments found on the gravel surface. One of the preserved coils was of a very heavy rope about five centimetres in diameter. (p.70)

All the cordage and canvas artifacts collected by the Beattie team are now located at the University of Alberta. These highly degraded pieces represent a challenge to the researcher who tries to identify them. They could possibly be made of bast fibres; historically, it is almost certain that they would be bast (Sanctuary, 1980). Some of these articles will be examined using various methods to try to identify them conclusively.

Justification

Although it is fairly certain that the Franklin Collection cordage and canvas artifacts are composed of bast fibres, it is very difficult to tell which plants these bast fibres are from without information from primary sources such as the provisions list from the Franklin Expedition. It is known that the predominant material for making rope, up until the late nineteenth century had been Indian hemp. Flax, on the other hand, was preferred for netmaking (Sanctuary, 1980).

A source from the nineteenth century states that it was impossible to distinguish coarse linen from cloth made of hemp (Anonymous, 1845). Catling and Grayson (1982), Hock (1942), and Kirby (1963) echoed this by pointing out the difficulty in telling one bast fibre from another when these fibres are new and even minimally processed. Kirby (1963) talks about the confusion over whether linen or ramie was used to wrap Egyptian mummies stating that there are grounds for confusion between the two. When bast fibres are "cottonized" (see p.5), they become even more alike than they are when minimally processed, particularly when examined without the aid of a microscope. Colour, lustre and feel can be almost identical. Bogle (1979) states that jute and hemp resemble one another very closely and are difficult to identify microscopically.

In order for microscopic identification of degraded fibres to be possible, their features which are both unique to the species and stable to degradation must remain . If various stains or swelling agents are required, the fibres must react in characteristic ways. King (1974) states that distinguishing degraded plant from animal fibres is usually a simple process, but there have been cases, such as the textiles from the site of Catal Hüyük in Anatolia, where that has not been possible. King goes on to say that plant fibres are difficult to identify because processes necessary to prepare them for spinning can damage or change the fibre structure.

Botanists most often use whole plants when making identifications. For a useful comparative collection of plant fibres to be made, not only must specimens of various levels of maturity be collected, but, theoretically, they should be processed in the various manners used historically. Comparisons should be made among flax fibres that are dew

or water retted, for instance. This could only very rarely be accomplished. Many plant fibres present in ancient artifacts have not been used in modern times (King, 1974).

Goodway (1987) cites cases where specific knowledge of fibre type enables the approximate dating of an artifact. Identification of the fibre content of artifacts can occasionally aid in determining the type of climate a place had or the trade routes of some civilizations at a particular time (Jakes & Sibley, 1983; Schaffer, 1981a). Unfortunately, textiles usually do not survive for very long unless conditions are appropriate for their preservation. Locations as diverse as salt mines, deserts, peat bogs or lake water can provide preserving environments for textiles. When they survive and are identifiable, they give valuable information. They can help to tell who manufactured, wore, used and sometimes discarded them. King (1974) cites the few writings she is aware of on textile, fibre or dye analysis in relation to archaeology. She says that there are few examples of fibre identification in Pre-Columbian pieces except in basketry, where whole stems or, at least, several different plant tissues are found.

Statement of Purpose and Objectives

Although many sources state how difficult it is to differentiate bast fibres from each other or from hard fibres in the leaves of monocotyledons, very few offer any solutions to the problem. There are chemical and other tests which do not conclusively identify a fibre, but rather, sometimes tell what it is not. These tests are not generally considered to be very reliable or precise. If the tests were used carefully, however, it is possible that consistency of results may be obtained. As well, the discrepancies in appearance and morphology within a single species and even within the fibres of an individual plant can lead to confusion and difficulty in the identification process.

Microscopically, bast fibres are remarkably alike. Almost all visible characteristics of fibres are present in every species commonly used in the past or present. The differences found in these fibres are a question of degree. They are very subtle.

There are three objectives of this research project. The first one is to develop a scheme which may make possible the conclusive identification of four bast fibres - flax, hemp, jute and ramie. It is hoped that this scheme will give a means of positively

identifying these fibres, however, discrepancies within a single species and among individual fibres may make absolute identification impossible. In order to accomplish this, a number of tests will be screened, then administered to known undegraded specimens.

The second objective is to determine whether there are characteristics which persist as flax, hemp, jute and ramie are degraded and, if present, to determine whether these traits are useful in identifying unknown fibres.

Finally, the series of tests will be used on several unknown and possibly degraded hard or soft fibres, including some specimens of the cordage and canvas from the Franklin Collection, to determine if they can be identified.

The purpose of this research is to begin to develop of a scheme for the positive identification of flax, hemp, jute and ramie, whether they are degraded or not. Ultimately, though beyond the scope of this project, is the development of a scheme for the positive identification of all unknown bast fibres, whether they are degraded or not. When the fibres are undegraded, their identities would be revealed conclusively. When degraded, if consistent species traits persist, the identification of the degraded fibres should be possible as well.

Four bast fibres, flax, hemp, jute and ramie, were chosen for study in order to limit the size of the project. Another reason they were chosen is because, historically, their cultivation and use go back several thousand years. They are also all still in wide use today (O'Shea, 1989; Quayyum & Terkelsen, 1989; Smyth, 1988).

Definitions

For the purpose of this study:

Actinomycetales An order of bacteria containing forms that develop a branched mycelium reminiscent of fungal growth forms, though on a far smaller scale (Blackmore & Tootill, 1988).

Bast fibre Originally phloem fibre, now any extraxylary fibre (Esau, 1977). In this study, bast fibre is used to mean phloem fibre.

Cambium An area which forms new cells by division. The products are formed by periclinal divisions commonly contributed in two directions and arranged in radial files. The term is preferably applied to the two lateral meristems, the vascular cambium and the cork cambium (Esau, 1977).

Cotyledon The first leaf or leaves of the embryo in seed plants (Blackmore & Tootill, 1988).

Cottonize To reduce a bast fibre to the fibre length of cotton and to make it capable of being used in the manufacture of articles resembling those made of cotton (Wingate, 1979). To process a bast fibre as a separated single cell, but not further (Menzi & Bigler, 1957).

Crossmarkings Parts of the attached walls from adjacent cells or the remaining impression left by adjacent cells after their removal (Catling and Grayson, 1982).

Decorticate Process of separating the bark and woody matter from bast fibres (Wingate, 1979).

Dicotyledon A seed plant having an embryo with two cotyledons. Other characteristics include having broad leaves with branching veins, flower parts in fours or fives, a persistent primary root developing into a taproot, and the vascular bundles arranged in a ring (Blackmore & Tootill, 1988).

Dislocations Regions in fibre cells where the cell wall has suffered damage by compression either during growth or after growth due to mechanical means (Catling and Grayson, 1982). They appear as I, V, or X-like markings or as swellings and are amorphous in nature. They are also called nodes.

Extraxylary fibres Fibres in various tissue regions other than the xylem (Esau, 1977).

Hard Fibre The fibres of the leaves of monocotyledons. They have strongly lignified walls and are hard and stiff (Esau, 1977).

Homopolymer A polymer composed of one kind of monomer (Mathews & VanHolde, 1990).

Lignin An organic substance or mixture of substances of high carbon content derived from phenylpropane and distinct from carbohydrates. Associated with cellulose in the walls of many cells (Esau, 1977).

Lumen (pl. lumina) The space bound by the cell wall (Esau, 1977).

Monocotyledon A seed plant having an embryo with one cotyledon. Other characteristics include having narrow parallel-veined leaves, flower parts in threes or multiples thereof, a fibrous root system, and numerous scattered vascular bundles (Blackmore & Tootill, 1988).

Monoecious The quality of having the female and male reproductive organs separated in different floral structures on the same plant (Blackmore & Tootill, 1988).

Panicle An inflorescence in which the flowers are formed on stalks arising alternately or spirally from the main axis (Blackmore & Tootill, 1988).

Nodes (See dislocations)

Parenchyma Tissue composed of parenchyma cells which are typically not distinctly specialized. They are concerned with one or more of the various physiological and biochemical activities in plants. They vary in size, form and wall structure (Esau, 1977).

Petiole Stalk of a leaf (Esau, 1977).

Phloem The principal food-conducting tissue of the vascular plant composed mainly of sieve elements, various kinds of parenchyma cells, fibres and sclereids (Esau, 1977).

Phyllotaxis The mode in which the leaves are arranged on the axis of a shoot (Esau, 1977).

Procambium An area which forms new cells by division. The cells differentiate into the primary vascular tissue (Esau, 1977).

Pseudomorph Mineralized textile fibres; a mineral having the characteristic outward form of something else (Jakes & Sibley, 1983).

Rhizome An underground stem that grows horizontally and, through branching, acts as an agent of vegetative propagation (Blackmore & Tootill, 1988).

Soft Fibre The phloem fibres of dicotyledons. They are usually less lignified and are soft and flexible relative to hard fibres (Esau, 1977).

Stoma (pl. stomata) An opening in the epidermis of leaves and stems bordered by two guard cells and serving in gas exchange (Esau, 1977).

Vascular cambium An area which forms cells of the secondary vascular tissues, secondary phloem and secondary xylem, in stem and root. It is located between those two tissues and, by periclinal divisions, gives off cells toward both tissues (Esau, 1977).

Xyloglucans A polysaccharide which is the primary component of hemicellulose in the primary cell wall (Albersheim, McNeil & Labavitch, 1977).

CHAPTER 2 REVIEW OF THE LITERATURE

Introduction

Bast fibres are extraxylary fibre cells of dicotyledons. Hard fibres are found in the leaves of monocotyledons and are considerably more lignified than the soft or bast fibres which are found in the stems of dicotyledons (Goodyear, 1971). Both fibre types provide support to various plant parts after elongation is complete (Catling & Grayson, 1982). They are known as ultimates in the textile industry and simply fibres in plant science (Maiti, 1980; Batra, 1985; Preston,1963). Fibre strands consist of groups of these long, tapering spindle-shaped cells cemented together with hemicelluloses, pectins and other materials (Maiti, 1979). Their ends overlap with each other forming continuous filaments throughout the stem (Basu, 1980a). These are the "fibres" of commerce (Esau, 1972; Batra, 1985). They are used for many purposes in industry, most notably for textiles and cordage.

Bast Fibre Plants

In soft fibre plants, the outermost layer of the stem is the epidermis (Figure 1). It is usually made up of a single cell layer. Next comes the cortex which is the ground tissue between the epidermis and the vascular system. The cambium is the site of cell division which lays down the phloem area where the bast fibre cells, or ultimates, are found in bundles amongst the phloem parenchyma cells. Flax and ramie fibres are primary in nature and as such are laid down by the procambium. Commercially used hemp and jute are secondary in nature and are a product of the vascular cambium (Kirby, 1963; Maiti,1979). All bast fibre cells have primary and secondary walls.

The number of fibre cells in a patch or bundle depends on species and variety. In flax and ramie, fibres are not arranged in pyramidal wedges as they are in hemp and jute, but are in smaller, more isolated groups (Basu, 1980a).

Height and basal diameter of stems in most bast fibre plants correlate positively with fibre yield (Basu, 1980a). As plants become more mature, however, the fibres become coarser. This is not desirable flax or in hemp that is to be used for garments. Ideally, each crop is harvested when it is at the stage of maturity appropriate to its end use. Seed

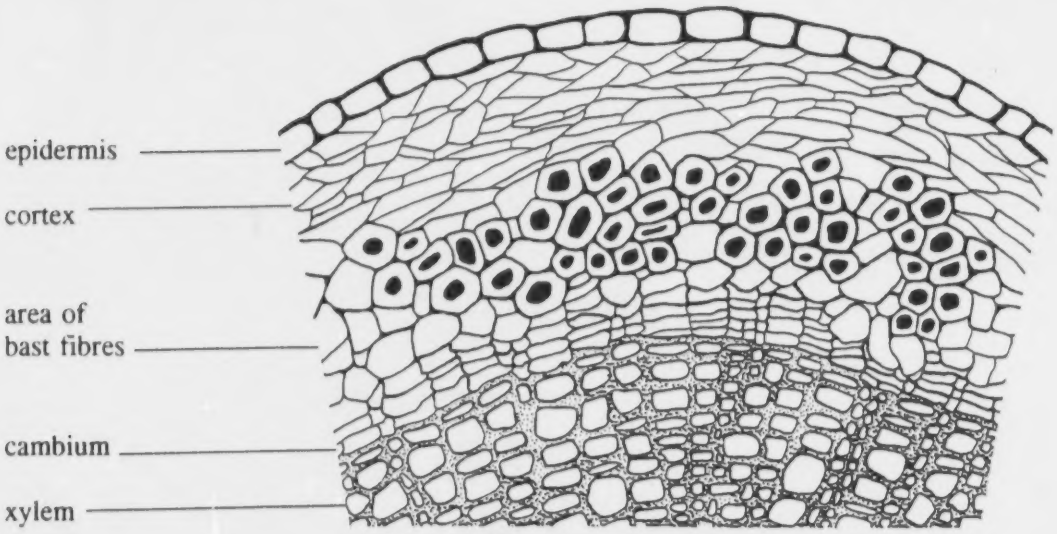


Figure 1. Transverse section of stem showing bast fibre cells.

harvesting necessitates plants being allowed to mature more fully than does most fibre harvesting.

Soft Fibres

Soft fibres consist of long cells with secondary cell walls that are thick compared to most plant cell secondary walls. They are called soft because, compared to hard fibres, they are indeed more soft and flexible. Once the fibre cells have completed elongation, they serve as support elements in the stem and thus, whether lignified or not, are relatively rigid compared to other cells in the plant. The degree of lignification in fibres can vary depending on the species and on the level of maturity (Esau, 1972), however, hard fibres generally are more lignified.

Growth

Soft fibre cells grow in both a coordinated and an intrusive fashion; their length increases along with adjacent cells while the adjacent cells are also lengthening, but the fibre continues to grow by apical intrusive growth at both ends. The fibre may be multi-

nucleate at this point with the intrusively growing parts of the cell having no pits. If, during intrusive growth, a fibre end is obstructed by another cell, it will grow alongside that cell. In so doing, it may be required to take on a curved, hooked or forked shape (Esau, 1972).

After the primary cell wall is laid down, secondary cell wall deposition in fibres occurs in the median area of the cell first, where coordinated growth has stopped. This secondary cell wall is deposited progressively in both directions to the cell tips following cessation of growth in these areas (Esau, 1972).

Fine Structure

As fibres are no longer living plant material once they are removed from the organism, there is no need to analyze the components of the living protoplast of the cell. Attention need only be paid to the cell wall, the middle lamella and any remnants of other types of cells still adhering to the fibres.

Cell walls in fibre cells occur in layers because of the way they are laid down. The outer layer, or primary cell wall is formed first and is followed by the secondary cell wall. The primary cell wall, particularly if there is a secondary cell wall present, is quite thin.

Soft fibre cell walls have few pits and these tend to occur near the middle of the cell when viewed longitudinally. According to Maiti (1980), the slit-like pits take on the direction of the cellulose microfibrils in the primary cell wall.

The thicker secondary cell wall consists of three layers from the outside in; the S1, the S2 and the S3 layers (Esau, 1977) and comprises 90% of the total weight of the fibre (Jakes & Sibley, 1983). The helical slope of the thin S1 layer of microfibrils makes a large angle with the long axis of the fibre (Figure 2). Therefore it is almost horizontal relative to the fibre axis. The much thicker S2 layer makes an angle which is small and so the helical slope is almost parallel to the fibre axis. The S3 layer is similar to the S1 layer (Esau, 1972). Batra (1985) states that microfibrils exist in series of layers. They spiral around the fibre axis in directions alternating from Z to S to Z, etc., repeatedly. This seems unlikely as without a more precise directional organization of the microfibrils

in the fibre cell walls, the results of the twist test would have no consistency (see Chapter 3). Authors generally agree that flax and ramie twist in one direction, while hemp and jute twist in the opposite direction to flax and ramie when the test is administered. Within the literature consulted many discrepancies were found regarding helical slope.

The cellulose framework of the fibre cell wall consists of a system of fibrils made up of closely packed cellulose molecules (Hollen & Saddler, 1964). The largest system, known as macrofibrils is composed of microfibrils. Macrofibrils can sometimes be seen with an optical microscope, while microfibrils necessitate the use of a scanning or transmission electron microscope. The more orderly, crystalline areas of the cellulose microfibrils are known as micelles. Other areas where the molecules are less orderly than the micelles are said to be amorphous (Esau, 1972). Preston (1963) states that microfibrils range from between about 80 Å and 250 Å in width, are virtually endless in length and are half as thick as they are wide. He goes on to say that the broader faces of the microfibrils lie parallel to the surface of the cell wall. At the same time the microfibrils can be found in a completely random arrangement in regard to each other, or range all the way to the almost completely parallel. The detailed structure of the cell wall can be seen in Figure 2.

The middle lamella is the area of union of the primary cell walls of two adjacent cells. It is mostly pectin but can contain a high percentage of lignin. In cells with secondary cell walls, the two adjacent primary cell walls and the middle lamella can appear as one layer (Esau, 1972).

Chemical Components

Approximately 2000 million years ago the process of photosynthesis is thought to have begun in primitive aquatic plant cells. These simple plants had no need for any rigid material in their walls. Not until the Devonian and Carboniferous periods (300 million years ago) did plants start to emerge from the water and stand upright. The requirement of exposure to light and to gases for absorption necessitated some sort of strengthening material such as the strong cellulose polymer (Linskens, 1989).

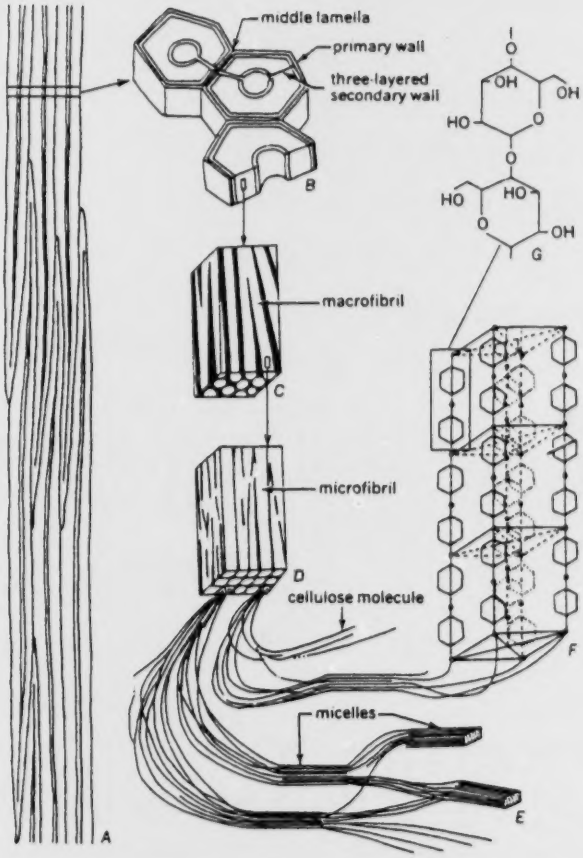


Figure 2 Detailed structure of cell walls. A, strand of fibre cells. B, cross section of fibre cells showing gross layering: a layer of primary wall and three layers of secondary wall. C, fragment from middle layer of secondary wall showing macrofibrils (white) of cellulose and interfibrillar spaces (black), which are filled with noncellulosic materials. D, fragment of a macrofibril showing microfibrils. the spaces among microfibrils (black) are filled with noncellulosic materials. E, structure of microfibrils: chainlike molecules of cellulose, which in some parts of microfibrils are orderly arranged. These parts are the micelles. F, fragment of a micelle showing parts of chainlike cellulose molecules arranged in a space lattice. G, two glucose residues connected by an oxygen atom - a fragment of a cellulose molecule.

Note. From *Anatomy of Seed Plants* (2nd ed.)(p.45) by Katherine Esau, 1977, New York: John Wiley & Sons. Reprinted by permission.

In growing plant cells, the cell walls are composed of 90% polysaccharides and 10% protein (Albersheim, McNeil & Labavitch, 1977). Three major classes of polysaccharides, which are polymers of simple sugars or saccharides, are present, namely cellulose, hemicelluloses and pectins (Fry, 1989).

Alpha-cellulose, also known as cellulose I, or simply cellulose makes up the first group. Cellulose forms the skeletal framework of the wall (Batra, 1985). It is a simple polymer composed of one kind of monomer and, therefore, is a homopolysaccharide. It is the most abundant polymer in the biosphere (Mathews & VanHolde, 1990). Two units of glucose combine to form the repeating unit called cellobiose, a disaccharide. These glucose residues are joined together by ß(1→4) linkages forming unbranched chains of great length with a strong tendency to self-associate into microfibrils (Hayashi, 1989; Sharples, 1963; Trotman, 1984). At least 99 per cent of the bonds between glucose monomers are of the ß(1→4) type (Peters, 1963). Only in beta linkages can the cellulose polymer be orientated in a straight line and thus be potentially fibre forming (Trotman, 1984) (Figure 3).

Preston (1963), believes that cellulose can contain a family of polysaccharides besides glucose. He says that when xylose is removed from cellulose, the degree of polymerization of cellulose is reduced, because the cellulose polymer chain has been broken, proving that glucose and xylose form a common structure. Trotman (1984), on the other hand, states that the cellulose polymer consists of nothing but glucose monomers.

In the self-association of cellulose polymers into microfibrils, hydrogen bonds form between the hydrogen of a hydroxyl group on one glucose unit and an oxygen on another unit. The bonds are individually weak, but collectively, strong, and there are many of them (Albersheim et al., 1977). While glucose is soluble in water, cellulose is not because of its high molecular weight and crystallinity. The hydrogen bonds, together with the characteristic linear and roughly planar shape of the cellulose molecule, allow it to pack together with other cellulose molecules and be bonded at many sites (Peters, 1963). Cellulose will dissolve in cuprammonium hydroxide and in concentrated solutions

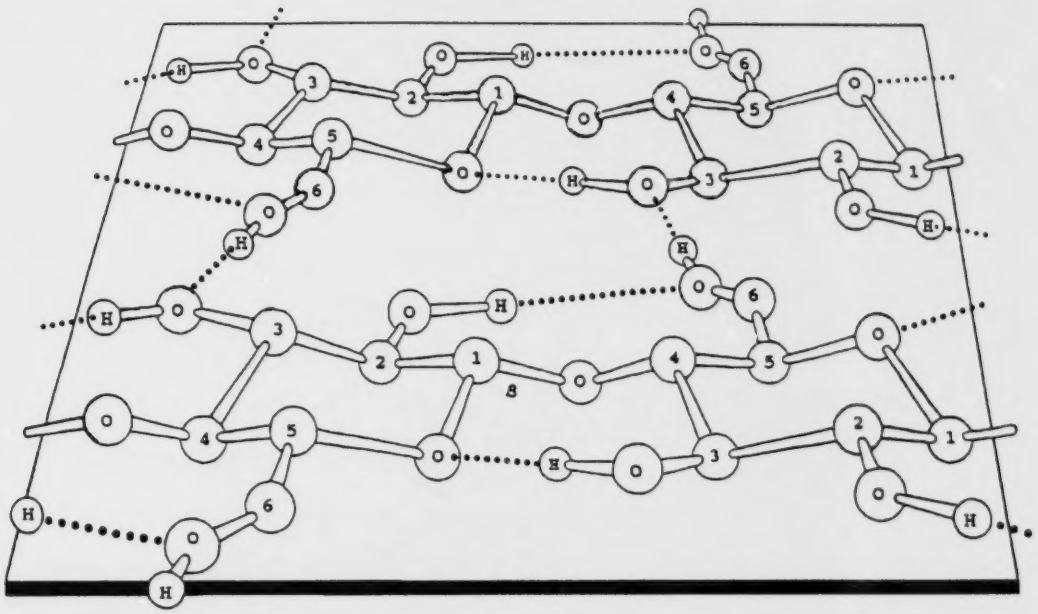


Figure 3. Planar cellulose structure showing ß(1→4) linkages and hydrogen bonds

of certain salts and some complexes of metals as well as ethylenediamine (Trotman, 1984).

The longer that cellulose polymers are, the greater the strength of the cell wall, but with limited extensibility (Mathews & VanHolde, 1990). Rigidity is imparted by the ring structure of glucose (Jakes & Sibley, 1983), and by hydrogen bonding. Molecules can reach 4μm in length (Esau, 1977).

Cellulose and starch differ only in their stereo chemistry. Starch has α(1→4) rather than ß(1→4) bonding, but this is sufficient to prevent cellulose bonds from being cleaved by certain enzymes that can cleave those in starch (Mathews & VanHolde, 1990).

Microfibrils of cellulose are embedded in an almost entirely amorphous matrix of hemicelluloses (Mathews & VanHolde, 1990; Feller, Lee & Curran,1985). Hemicelluloses, the second major group of polysaccharides, which are rigid and rod shaped, hydrogen bond to the hydroxyl groups of the cellulose (Fry, 1989). They are

most often branched molecules with a lower molecular weight than cellulose (Feller et al., 1985). Some hemicelluloses act as a cross-link between non-cellulosic polymers and cellulose (Esau, 1972). They are insoluble in water and soluble in cold and hot alkali while being hydrolysed in acids more easily then cellulose, thus yielding monosaccharides (Feller et al., 1985). Most hemicelluloses are extractable with alkali (Preston, 1963). Xyloglucan is the most dominant hemicellulose in dicotyledon plant cells (Albersheim et al., 1977).

According to Florian (1990), hemicelluloses "act as a protective hydrated amorphous matrix surrounding cellulose fibrils" (p. 26), causing the areas between microfibrils to increase because of swelling. This flexible gel allows "strong crystalline regions to slide, compress and move away from each other when the cell is put under tension, compression or other load" (p. 26).

Pectins, the third group of polysaccharides, are jelly-like and acidic consisting of methylated polygalacturonic acid associated with galactose and arabinose (Preston, 1963). They do not bind nearly as tightly to the cellulose framework as do the hemicelluloses (Fry, 1989), and are found mostly in the middle lamella (Moore, 1989). Galactose and arabinose are homopolymers found attached to each other, but the precise configuration of attachment is not known (Albersheim et al., 1977). The presence of pectins is associated with a high degree of swelling in water (Preston, 1963).

The chemistry of the hemicelluloses and pectins is far more complex than that of cellulose. Both are composed of two or more kinds of sugar units which are connected by several kinds of glycosidic linkages. These polysaccharides are bonded covalently to one another (Albersheim et al., 1977). They can not form long chain polymers, however, and so can not form crystalline areas (Kronkright, 1990).

In addition to the above discussed polysaccharides, there is another material of great importance in the building of some cell walls. Lignin is a complex, heterogenous polymer of phenylpropanoid units (Esau, 1972). "...Its chemical structure is known only in the form of a structural average of several characteristic intermolecular linkages..." (Umezawa & Higuchi, 1989, p.161). It is aromatic in character, containing a large proportion of methoxyl and phenolic hydroxyl groups. It is amorphous in structure (Jakes

& Sibley, 1983). Depending on the type of cell, the age of a plant, and the species of a plant, lignin can be found to a lesser or greater extent impregnating the cellulose between the microfibrils in the cell wall (Preston, 1963). Within a species a cell will become more lignified as it ages. According to Esau (1972), lignin links chemically to polysaccharides, filling in the spaces within the fibrillar cellulose framework. Sharples (1963), however, states that chemically bound impurities in cellulose do not exist to an extent of more than one per cent. He says that the precise relationship between lignin as well as between hemicelluloses and cellulose still remains problematic. Both Mathews (1952) and Weindling (1947) call this combination of cellulose and lignin "lignocellulose" or "bastose".

With different species, the amount of lignin in a fibre varies. Flax fibre, for instance, will have no lignin or very little unless it has been allowed to mature past the point of being commercially useable. Jute fibre, however, is always found to be lignified in the usable state. Rahman (1978), states:

> The cellulosic and non-cellulosic constituents [in jute] are present as two interpenetrating systems in the primary and secondary walls, and the middle lamella is composed mainly of lignin and a trace of pectin. The lignin penetrates the cellulose like a system of 'nerves', middle lamellar substances being the 'central nerve'. (p. 287)

Cellulose and lignin form the major structural components of plant cells (Linskens, 1989). Lignin is found in its purest form in the middle lamella, but most abundantly in the secondary cell wall (Kawahara, 1989).

An important property of lignin is its sensitivity to ultraviolet light. It oxidizes under sunlight and turns yellow. As the percentage of lignin is reduced in a fibre during processing, so too is that fibre's potential for degradation (Textile conservation Notes No. 5).

One last ingredient can sometimes be found in bast fibre cells. As these cells are no longer living, or functioning, protoplasmic contents are not important. Remnants of protoplasm can often remain in cell lumina, however, even in processed fibres. These generally are nitrogenous in nature (Batra, 1985).

Flax History and Uses

Flax is thought to have originated somewhere in alluvial soil in the Caucasus. It was the earliest vegetable fibre to be spun (Basu, 1981). The oldest surviving cloth in Western Europe which was found at the site of Neolithic lake dwellings in Switzerland is linen from c.2000 B.C. The long combed fibres known as line are of higher quality than the broken, shorter, more tangled fibres called tow (Hollen & Saddler, 1964). When flax fibre is processed into yarn and then subsequently cloth, it becomes linen.

Joseph (1986) states that very fine linen cloth with a fabric count of up to 500 threads per inch was woven in Egypt as far back as 2500 B.C. Some of it was almost transparent. The Royal Ontario Museum has some Egyptian linen that was produced c.1450-2000 B.C. Paper made of linen rag was being made in China in 105 A.D. Flax was also used in rope-making in 480 A.D. In the seventeenth century, Ireland became a linen producing centre. A group of Huguenot weavers settled there after fleeing persecution in France (Hollen & Saddler, 1964). The highest quality linen produced in England in the 1840s was made from flax grown in Holland (A day in a Leeds flax mill, 1843).

Flax is grown in North and South Temperate Zones. For some time before World War I, nearly 90 per cent of the world's production was from Russia (Johnson, 1927). More recently, Russia has been producing about 75 per cent of the world's output (Basu, 1981). Belgian linen, retted in the river Lys, was considered to be some of the highest in quality. In 1943, retting in the Lys was forbidden by royal decree as acidic pollution caused by the process became excessive (Dujardin, 1948). Flax is used mostly for clothing and household textiles, thread, cordage, nets and high quality paper. Once, all European shoes were sown with it (Smyth, 1988). Since 1980, linen has experienced a resurgence in popularity.

Plant Characteristics

The flax family (*Linaceae*), consists of nine genera and 200 species of which *Linum usitatissimum*, the flax fibre plant, is one (Lawrence, 1969). It is the only species in the

family of importance in the commercial production of fibre (Kirby, 1963). *Linum usitatissimum* will be referred to hereafter as flax.

Flax is an annual. The leaves are alternate or opposite and rarely whorled (Lawrence, 1969), but according to Meicenheimer (1986), seedling leaves are opposite changing either to alternate at node two above the cotyledons or producing as many as twelve opposite nodes before becoming alternate. The leaves are attenuated, narrow and tapering at both ends, smooth and greyish green in colour (Basu, 1981). Once plants are established, they display a "closed" type of vascular system, having one sympodium (Meicenheimer, 1986). The flowers of the flax family are bisexual, and radially symmetric (Lawrence, 1969).

Flax grows from 1 to 1.3 m high. Each plant stem is 4 to 5 mm in diameter (Catling & Grayson, 1982). Flowering can occur during one short period or it can happen two or three times, depending on genetic or environmental factors (Hovland & Dybing, 1973). The plants are grown close together and must be harvested at the right time. If pulled too soon, the crop is scanty and weak, but if too late, the fibres become lignified and are harder to separate from other tissue. Flax stems must go through the retting process to free the fibrous bundles (Chase, 1982) (see chapter on processing). Further processing of the fibres must take place if the end products of spun yarn and linen cloth are to be realized, but without first retting, none of these more advanced steps can be performed.

According to Maiti (1980), flax fibre cells can occur either singly or in bundles in the stem. Even when found in groups, quite often by the time all processes such as bleaching and alkali treatments have taken place, each cell will be separate in a textile (Heyn, 1954).

Morphology

According to Catling and Grayson (1982), the length range of the flax fibre cell is from 1.6 to 24.0 mm ; its diameter range is from 11.7 to 32.0 μm (see Table 1 for other authors' observations). Diameters tend to be uniform in the central portion of the cell, tapering gradually to fine points (Strellis & Kennedy, 1967). Immature flax fibre cells

are oval in transverse section and the cell walls are thinner than in the mature fibre cell. Consequently, the lumen is relatively larger in the immature cell than in the mature cell (Cook, 1984). The cell walls in mature fibre cells are occasionally striated and the lumina narrow and regular. The cells are strongly polygonal to circular in transverse section (Catling & Grayson, 1982, Naslund, Vuong & Chanzy, 1988 & Batra, 1985), though each fibre cell bundle shows gradation in cell wall thickness and lumen size with the oldest cells adjacent to the cortex having the thickest walls and the narrowest lumen diameters (Fraser, Courtney & Harvey, 1982). Sometimes the lumen is difficult to see in a mature fibre cell because of the thickness of the cell wall (Maiti, 1980).

Flax fibre cells have dislocations in the shape of the letters I, X, or V which often bulge out from the surface of the cell. They are also called nodes (Strellis & Kennedy, 1967; Anderson, 1927). While many authors disagree about what causes dislocations, Catling and Grayson (1982) state that they are caused by compression, whether during growth, or afterward, during mechanical processing. Cross markings from adjacent cells such as parenchyma and xylem elements may be present very occasionally. There have been no reports of crystals enclosed in flax fibre cells (Catling & Grayson, 1982). Further information by various authors on the characteristics of flax fibre cells can be found in the tables following this chapter.

The outermost fibre cells of the flax plant stem are the first to develop secondary walls. These begin forming in the primary bast fibre cells after the area of the stem in which they occur ceases to elongate (Esau, 1943; Fraser et al., 1982). This allows for a greater number of cell wall lamellae in the fibres closer to the base of the stem than at the tip. Initially, the thickening of the secondary wall is rapid. It is then followed by a more gradual increase (Hock, 1942).

Lignification, which results in a reduction of silkiness, lustre and elasticity, starts in the area of the stem closest to the lower leaves when the plants are 27 to 35 cm high and four to six weeks old. According to Sharma (1986), it starts before flowering and increases up the stem with age. Fraser et al. (1982), however, state that it is initiated at the same time as flowering and progresses rapidly thereafter.

Fine Structure

The flax fibre cell walls, with their extensive secondary thickenings, contain a large amount of cellulose and usually a very small amount of lignin. They are approximately 90 per cent crystalline, except in the dislocations which are considerably more amorphous (Jakes & Sibley, 1983). The cellulose occurs in the form of crystalline microfibrils (see Figure 2).

As stated earlier, authors vary greatly in their views on the helical slopes of the cellulose microfbrils in the various layers of the cell wall. Hock (1942) and Jakes and Sibley (1983) state that a Z-twist is apparent in the outermost microfibrils and an S-twist in the layers beneath though Anderson (1926) states that each microfibrillar layer varies in orientation. Recently, Naslund et al. (1988) have reported finding that both flax and ramie have microfibrils which are oriented parallel to the fibre axis.

Environmental Effects on Flax Fibre

After processing, flax fibre contains very little or no lignin. It is, therefore, not particularly sensitive to ultraviolet light but vulnerable to microorganisms such as mildew (Goodyear, 1971). It becomes stronger when wet, having a moisture regain of 7% both unbleached and bleached under the standard conditions of 65% relative humidity and 70°F (Batra, 1985). While flax can be vulnerable to reduction, oxidation and thermal degradation, it is considered to be resistant to deterioration caused by age when compared to more amorphous fibres (Textile Conservation Notes No. 5).

Hemp History and Uses

Hemp, *Cannabis sativa*, is a native of Iran and India. It is a member of the mulberry (*Moraceae*) family and closely related to the nettle (*Urticaceae*) family (Lawrence, 1968). Joseph (1986) states that it was cultivated in China before 2,300 B.C. It has been used for rope and fabric making in parts of Asia and the Near East since pre-historic times (Aranyanak, 1991). It is grown in many places and in most climatic zones. Most often, it is made into very strong cordage and oakum, which is used to make boats and buildings watertight (Harris, 1954). Russian grown hemp was imported to Great

Britain in the eighteenth and nineteenth centuries for the rope and cordage industries (A day at a Leeds flax mill, 1842). *Very fine hemp cloth, often confused with linen, has been produced in Italy (Cook, 1984).* *C. sativa* will be referred to hereafter as hemp.

Plant Characteristics

The hemp plant is an annual and is the same plant that produces the narcotic drug marijuana. When used for fibre purposes, the plants are grown closely together and do not branch (Kirby, 1963). The male and female flowers occur on separate plants (The hemp plant, 1962).

The plants grow from 3 to 4.5 m high. The stems and leaves are very slightly hairy, while the stems are also ridged (Catling & Grayson, 1982). The leaves are palmately compound with from 7 to 11 leaflets (Ash, 1948; Kirby, 1963). Hemp must go through the retting process to obtain useable fibres (see chapter on processing). The strands produced consist of bundles of fibres (The Textile Institute, 1975). There may be between 2 and 20 fibres in a bundle (The hemp plant, 1962).

Morphology and Fine Fibre Structure

The length range of the hemp fibre cell is from 1.0 to 34.0 mm. Its diameter, often uneven throughout the cell, ranges from 16.3 to 67.1 μm (Catling & Grayson 1982) (see table 1 for other authors' observations). In transverse section, it is polygonal with rounded edges or it is cylindrical (The hemp plant, 1962; Batra, 1985). The cell walls of hemp are striated, with the fibrils of the primary cell wall twisting in a Z direction (The Textile Institute, 1975). The lumen is most commonly three to five times the width of the cell wall. It is broad, flat and narrows toward the end of the cell (Strellis & Kennedy, 1967). Slit-like pits, which are parallel to the long axis of the cell and which can coalesce, are sometimes visible with an optical microscope. The dislocations in hemp are similar to those in flax. Crossmarkings, which are found more frequently than in flax, are the resulting marks left from adjacent cells. There can be several marks on one fibre cell with the remains of other cells such as parenchyma attached to them (Mathews, 1952; Catling & Grayson, 1982). The fibre cell ends of hemp are blunt, thick-walled and

occasionally forked (Basu, 1981). Often other elements of the stem occur with hemp fibre cells. Tissues such as hairs and epidermis, as well as xylem elements and cluster crystals can be found. There are crystals present in ashed specimens of the fibre except when boiling or bleaching has taken place. Cluster crystals in short chains are often found and very occasionally, cubic or rhombic crystals (Catling & Grayson, 1982).

According to Kundu (1942), lignin is deposited in the middle lamella and the primary cell wall of hemp fibre cells. There may be some in the secondary cell wall as well. Kundu (1942) goes on to say that lignin can still be present in the primary cell wall of the fibre cells after processing. Kawahara (1989), however, believes that lignin is found most abundantly in the secondary cell wall of all bast fibre cells. Further information by various authors on the characteristics of hemp fibre cells can be found in the tables following this chapter.

Environmental Effects on Hemp Fibre

Processed hemp contains more lignin than flax, but much less than jute. Exact figures vary. It is consequently slightly vulnerable to degradation by ultraviolet light. Though having a moisture regain of 8% when both unbleached and bleached under the standard conditions of 65% relative humidity and at 70°F (Batra, 1985), the fibre nonetheless resists microbial attack. Relative humidity below 50 to 55% causes brittleness in the fibre (Textile Conservation Notes #5).

Jute History and Uses

Jute, *Corchorus capsularis*, (white jute) and *C. olitorius*, (Tossa jute), is a sub-tropical herbaceous annual belonging to the *Tiliaceae* family (Basu, 1980b). It is grown in India, Bangladesh, Nepal and Burma. It was cultivated extensively in the nineteenth century to supply the jute industries of Europe. Later, manufacturing took place in the country of origin (Quayyum & Terkelsen, 1989). Unlike flax and hemp, the jute crop must be cut down, not pulled (Mathews, 1952). It is the most important bast fibre on a tonnage basis, being greater than that of all other bast fibres put together (Cook, 1984). Jute is primarily used to make packaging materials such as burlap and for

carpet backings. Products such as carpet yarn, cordage, felts and paddings are also made from jute (Quayyum & Terkelsen, 1989). *Corchorus* will be referred to hereafter as jute.

Plant Characteristics

The jute plant is a woody stemmed annual which takes from three to five months to mature. It can grow from 2.5 to 6 m high (Catling & Grayson, 1982). The main differences between *C. capsularis* and *C. olitorius* are that in *C. capsularis*, the fruit is a globular pod with large chocolate brown seeds. The leaves have a bitter taste. In *C. olitorius*, the fruit is cylindrical with smaller, bluish-green, grey or black seeds. The leaves have a shiny upper surface, a rougher under surface and are almost tasteless (Kirby, 1963). Jute has small yellow flowers that occur in condensed cymes (Basu, 1980b). It must go through the retting process to be utilized (see chapter on processing).

Morphology

Jute fibre cells are arranged together in bundles of from 12 to 25 cells (Kundu, 1942). The length range of the jute fibre cell is from 0.6 to 5.3 mm making it relatively short compared to other bast fibres. It has slender, pointed ends and is polygonal with sharply defined angles and little variation in diameter in transverse section. Its diameter range is from 9.30 to 32.6 μm having irregularly thickened walls and a well defined oval or round lumen (Catling & Grayson, 1982; Menzi & Bigler, 1957) (see Table 1 for other authors).

In transverse section, jute lumina vary in width from being very wide to just a slit, and they can be interrupted. According to Mathews, (1952), they broaden out toward the ends of the fibre cells, causing the cell walls to become very thin. The pits on the cell walls are bordered and funnel-shaped in side view (Catling & Grayson, 1982). Catling and Grayson (1982) state that there are only a few faint cross markings or often none, but according to Strellis and Kennedy (1967) dislocations and cross-markings are more numerous if mechanical treatment is rough. There is little other material besides fibre cells present, although sometimes parenchyma is in evidence. Single cluster crystals

are present in the ash (Catling & Grayson, 1982). Jute fibre cells and the surrounding middle lamella are highly lignified (Strelis & Kennedy, 1967).

The hemicelluloses that cement the cells together are not completely formed in immature fibres so that complete lateral cohesion does not take place immediately. According to Srivastava and Adams (1959), mature jute is 30% hemicellulose. No pectin remains in well cleaned jute fibre, but often poor retting allows for some residual material, including pectin (Kundu, Basak & Sarcar, 1959).

Fine Structure

The highly oriented fibrillar arrangement of the secondary wall of the jute fibre cell is enveloped in the Z-twisted fibrillar envelope of the primary wall (Hock, 1942, Sen & De, 1949). Mukhopadhyay, Bandyopadhyay and Mukhopadhyay (1985), however, do not concur with this finding. They use the words wall and layer interchangeably and say that the primary layer generally has fibrils which are criss-cross in arrangement. The fibrils in the secondary layer occur in straight layers, almost parallel to the fibre axis with some Z-twisted fibrils underneath followed by more straight fibrils. They also say that, with the odd exception, nodes are present only in the primary layer. In immature fibre cells, the fibrils of the secondary layer are originally aligned at an angle to the fibre cell axis which decreases with growth. Chemically, jute has a natural affinity for basic dyes. Its acidity is due to the presence of uronic acid (Kundu et al. 1959).

Environmental Effects on Jute Fibre

Because of the high degree of lignin present in jute fibre cells, they are extremely vulnerable to chemical degradation under ultraviolet light. At the same time, the lignin helps protect cellulose from attack by microorganisms (Goodyear, 1971). Another aspect in the deterioration of jute is water absorbency. Under the standard conditions of 65% relative humidity and 70°F, jute has a moisture regain of 12% if unbleached and 10 % if bleached (Batra, 1985). It still becomes vulnerable to damage from microorganisms in spite of its lignin content. It also loses a high percentage of its strength when wet. Being a brittle fibre like hemp, it is recommended that it be kept at a relative humidity of at

least 50 to 55%. It is sensitive to both acidic and basic conditions. Jute, overall, does not resist deterioration well (Textile Conservation Notes #5).

Ramie History and Uses

Ramie, *Boehmeria nivea*, is grown in many countries including the United States and China. Hunan province in China, where it has been grown for thousands of years, is the biggest producer. This exceptionally lustrous fibre is the strongest vegetable fibre (Basu, 1981). Current uses include upholstery, tablecloths, napkins, curtains, clothing and high grade paper. In Switzerland, it has been used in combination with polypropylene as a geotextile in the reconstruction of river banks (O'Shea, 1989).

The single fibre cells of ramie represent the fibre strands of commerce (Maiti, 1979). Menzi and Bigler (1957), state that ramie is almost always processed as separate single fibres and not single cells joined end to end. According to them this is cottonization (See p. 5).

Plant Characteristics

The nettle family (*Urticaceae*), consists of approximately 42 genera and nearly 600 species. Most species are tropical or subtropical requiring a warm, humid climate. There are 80 species of *Boehmeria* of which *B. nivea* is one (Lawrence, 1969). *B. nivea* will be referred to, hereafter, as ramie.

Ramie, a perennial produces three or four crops each year. The straight, striated canes grow from 1.2 to 2.4 m high with a stem diameter of from 10 to 20 cm (O'Shea, 1989). They develop from underground rhizomes which run laterally from the parent plant (Basu, 1981). Ramie is the only fibre of the four in this study which does not go through the retting process (see chapter on processing).

The leaves of ramie grow on long petioles and are covered with inconspicuous hairs. They are round or heart-shaped, 10 to 20 cm wide, with dark green surfaces and finely serrated margins. In some varieties of ramie the undersides of the leaves are white and felt-like. Ramie has no stinging hairs unlike many other members of the family. The plant is monoecious and female flowers are developed on upper parts of the stem while

the earlier maturing male flowers are found on the lower parts (Kirby, 1963). The flowers of ramie occur in axillary panicles in clusters and are small and greenish white in colour. Small light brown fruits are produced which contain tiny black coloured seeds. The rhizomes rather than the seeds are used for propagating ramie (Basu, 1981).

Morphology

Ramie fibre cells possess very thin primary cell walls. The secondary cell walls have three layers which can be seen with the polarizing microscope (Ray, 1975). According to Kundu and Sen (1960) secondary wall deposition occurs simultaneously with the extension of the fibre cells. This does not concur with Esau (1972) who states that secondary cell wall deposition occurs after growth is completed in that area of the cell.

Ramie fibre cells have an angular outline, in transverse section, when young (Kundu & Sen, 1960); however, later they become oval or round and sometimes flat. There can be dislocations or nodes and a tendency for radial cracks to develop (Batra, 1985). Longitudinal striations are frequent (The Textile Institute, 1975). The rounded points of the fibre ends are thick-walled with the lumen sometimes reduced to a line (Mathews, 1952).

The length range of the ramie fibre cell is from 13.0 to 82.7 mm, being by far the longest fibre of the four selected for this study (Catling & Grayson, 1982). Aldaba (1927), claims to have isolated ramie fibre cells in the range of 400 to 550 mm from macerations. Its diameter range is from 12.5 to 65.9 μm according to Catling and Grayson (1982) and can vary within one cell (see Table 1 for other authors). Kundu and Sen (1960) state that the basal end of the cell is wider than the apical tip. The cell walls have elongated, slit-like pits parallel to the long axis of the cell, sometimes coalescing. The lumina can be difficult to see because of striations in the cell walls and the great tangling of the fibre cells. In spite of thick cell walls, lumina are usually two to three times the width of the cell wall (Catling & Grayson, 1982; Maiti, 1980) and can contain granular material.

Maiti (1980) claims that the blunt fibre cell ends, larger regular lumen and highly thickened cell wall of ramie are unlike any other vegetable fibre. Though ramie may be

a very distinct fibre, other authors give different information on the appearances of these three characteristics.

Dislocations, similar to those in flax, are a common occurrence in ramie as are fine cross markings which often have the remains of parenchyma cells still attached. The crossmarkings are finer than in flax. There can be occasional marks of chambered cells. Very little hair and vessel tissue is found with processed fibre cells. Parenchyma and free cluster crystals or cluster crystals in chambered cells are frequent features (Catling & Grayson, 1982; Kerr, 1988). Further information by various authors on the characteristics of ramie fibre cells can be found in the tables following this chapter.

Fine Structure

According to Naslund et. al. (1988), ramie and flax are similar, in that their cellulose microfibrils are oriented nearly parallel to the fibre cell axis. Heyn (1954) considers ramie's microfibrils to be completely parallel to the fibre cell axis. The gum in decorticated ramie stems (see chapter on processing) is a polysaccharide and is found mostly in the fibrous area, but not actually in the crystalline structure of the cellulose. This is similar to the behavior of hemicelluloses in jute. Removal of the gum enables the cellulose crystallites to attain a higher degree of perfection in the crystalline order. However, at the same time, alignment of the crystallites along the fibre cell axis deteriorates (Ray, Bag & Chakravarty, 1975). Ramie fibre cells have the highest cellulose content of all vegetable fibres at approximately 84% according to Basu (1981) and at 91% according to Harris (1954).

Environmental Effects on Ramie Fibre

Ramie fibre has an extremely low percentage of lignin (0.6%) and a moisture regain of 6% when bleached at 65% relative humidity at 70°F (Batra, 1985). It is not particularly light sensitive and, like flax, is stronger when wet. Except for a tendency to mildew, generally, ramie has good ability to resist deterioration well over long periods of time (Goodyear, 1971; Bogle, 1979).

Comparisons of Flax, Hemp, Jute and Ramie1

A great deal has been written about the characteristics of bast fibres. Information on such things as fibre cell length and diameter, pitting, crystals, and wall thickness are just a few of the characteristics which have been reported by many authors.

The problems with the wealth of information on bast fibre cell characteristics are the great inconsistencies within that information. Not only is there a difference between authors in what they see, but there is inconsistency in terminology. Measurements also vary widely. It is tempting to attribute the great variety of information to poor data collection methods, and indeed, this may sometimes be the case. Often, another reason is the cause. As stated previously, the diversity among fibre cells from one plant can be as great as the diversity between fibre cells from two plants of the same species and even between species. The differences in characteristics reported by researchers are most often simply due to the vast differences among fibre cells.

Detecting the differences among vegetable fibre cells can also be difficult because of the great sensitivity of morphological features to factors such as growing conditions, level of maturity, location in the leaf or stem, the type of processing and degree of degradation. There are some basic differences between leaf and stem fibre cells that are detectable in transverse sections of cell bundles. Stem, or soft fibre cells, occur in bundles of less than 30 cells and the bundle shapes are irregular. Leaf, or hard fibres can be found in bundles of up to 100 cells and the bundle shapes can be circular, elliptical or crescent shaped (Schaffer, 1981b).

It is possible to find differences among the four fibres in this study in terms of appearance to the naked eye, as well as in morphology and fine structure. A comparison based on fibre cells in the undegraded state, after basic processing is given.

Flax fibre, which is approximately 76% cellulose (Harris, 1954), is fine and silky. According to Basu (1981), it is 64% cellulose. It ranges from white to grey to light brown in colour and is easily bleached to white. There are usually 12 to 14 fibre cells per bundle according to Landi (1985) and Florian (1990), while Mathews (1952) says they are often found singly and there are seldom more than three or four fibre cells per bundle.

Hemp is approximately 77% cellulose (Harris, 1954), and is stiff and harsh compared to flax (Rice, 1963). The fibre cells are arranged in pyramidal wedges in the stem (Basu, 1980b). It is grey or dark brown and is difficult to bleach (Joseph, 1986). It is stronger than flax and does not rot as readily as jute in water (Johnson, 1927). Bundle cell numbers are similar to flax (Landi, 1985).

Jute is approximately 63% cellulose (Harris, 1954), having the lowest cellulosic content of the four fibres. The fibre cells are arranged in pyramidal wedges in the stem (Basu, 1980). Jute can be lustrous and is a golden yellow, sometimes with a pink or reddish tinge. It is difficult to bleach to white and to dye (Joseph, 1986). The fibre cell is weak compared to flax or hemp and is very short. It is not durable when exposed to dampness (Johnson, 1927). It deteriorates rapidly under sunlight and darkens in colour. Jute is sensitive to acids and alkalis (Bogle, 1979). There are usually 25 to 50 fibre cells per bundle (Landi, 1985).

Ramie fibre, at 91% cellulose (Harris, 1954) has by far the highest per cent of cellulose of the four fibres and also has the longest fibre cells. Basu (1981) claims that ramie is 84% cellulose and that with practically no lignification is the purest of all cellulose fibres. It is very white and highly lustrous (Hearle & Peters, 1963). Ramie is not deteriorated by exposure to moisture and is stronger than flax (Johnson, 1927). Fibre bundles are very small and can consist of just single cells (Mathews, 1952).

The following tables are a compilation of observations and measurements taken by many authors. Table 1 gives comparisons of fibre diameters. Table 2 is concerned with descriptions giving fibre shapes and characteristics. Table 3 describes lumina and cell walls in the four fibres. Table 4 describes dislocations while Table 5 describes cell ends. Degree of lignin present in the fibres is discussed in Table 6.

Many discrepancies and duplications of information can be found in the tables. Their usefulness is in both using them to view the differences between authors and as a reference when observations are made on fibre specimens.

Table 1
Fibre Cell Diameters (µm)

Species	Authors			
	Catling & Grayson (1982)	Florian (1990)	Heyn (1954)	Peters (1963)
Flax	Range 11.68 to 31.96 mean 19	5 to 38	Much smaller than ramie	10 to 30
Hemp	Range 16.27 to 67.10 mean 30	10 to 15	Much greater variety than with flax	-
Jute		12 to 18	15 to 25	-
C. olitorius	Range 9.30 to 32.60, mean 20			
C. capsularis	Range 9.64 to 26.32, mean 18			
Ramie	Range 12.50 to 65.89 mean 31	25 to 75	40 to 80, largest diameter of all vegetable fibre cells	-

Table 1 (cont'd)
Fibre Cell Diameter (µm)

Species	Authors		
	Kundu (1942)	Mathews (1952)	Strellis & Kennedy (1967)
Flax	-	-	-
Hemp	34.2 (average)	Uneven diameter	Varies throughout length
Jute	-	-	-
Ramie	-	Frequently very broad and ribbon-like, never twisted, up to 80	Varies throughout length, larger in all dimensions than flax or hemp

Table 2
Fibre Cell Shape and Characteristics - Longitudinal

Species	Authors			
	Florian (1990)	Kundu & Sen (1960)	Mathews (1952)	Menzi & Bigler (1957)
Flax	Constant width throughout length	-	-	-
Hemp	Variation in width of cell	-	Longitudinal fractures and swollen fissures, occasional transverse markings, numerous striations	-
Jute	Constant width throughout length	-	-	-
Ramie	Widest commercial bast fibre cells, thickness varies throughout length	Cylindrical, many transverse markings	Transverse fissures, uneven diameter over cell length, can have heavy striations	Longitudinal and transverse fissures

Table 2 (cont'd)
Fibre Cell Shape and Characteristics - Longitudinal

Species	Authors		
	Strellis & Kennedy (1967)	Heyn (1954)	Koch (1963)
Flax	Cylindrical smooth surface	-	Smooth and cylindrical
Hemp	Cylindrical surface, striations and transverse fractures, swollen fissures	-	Smooth and cylindrical
Jute	Cylindrical little variation in diameter, smooth	Fibre cells taper gradually to a point at both ends	Cylindrical
Ramie	Frequent striations, finer than in flax	Shape of flat ribbon with twisted parts, like cotton, but more gradual	Very irregular, and extremely broad, has points of twisting

Table 3
Lumen and Cell Wall Characteristics

Species	Authors			
	Anderson (1927)	Bag, Ray, Das & Mukerjee (1987)	Catling & Grayson (1982)	Garner (1966)
Flax	Each layer of C.W. has striations and direction varies in successive layers (is reversed), no. of lamellae varies	-	C.W. occasionally striated, C.W. thick, lumen narrow, regular	Narrow lumen
Hemp	-	-	C.W. striated, lumen most commonly 3 to 5 times width of C.W.	Broad indistinct lumen, flat, narrows near end
Jute	-	Surface fibrils in fibres with high lustre are almost ∥ fibre axis, those on surface of fibres with poor lustre are not as parallel or clearly defined	Lumen of varying width, often varying regularly along whole length of cell	Broad variable lumen
Ramie	-	-	Lumen difficult to see (a)it varies (b)C.W. is striated (c)fibre cells tangle,lumen commonly 2 to 3 times width of C.W.	-

Note C.W. means cell wall.

Table 3 (cont'd)
Lumen and Cell Wall Characteristics

Species	Authors			
	Hock (1942)	Jakes & Sibley (1983)	Kundu & Sen (1960)	Maiti (1979)
Flax	Wall thicker in cells at base of stem than at tip, becomes thicker with maturity, can have from 22 to 30 lamellae, mostly ∥ axis, Z twist in outer layer, S in layers underneath	Fibrils spiral in Z twist in 1°C.W., S twist in 2°C.W., angle of inclination of spirals is 5°, no reversal of spirals, approximately 90% crystalline, small lumen	-	Hard to see lumen because of thickness of C.W.
Hemp	-	-	-	-
Jute	-	-	Fibrils highly oriented, 2°C.W. enveloped in sheath of flat Z twisted fibrillar wrapping of 1°C.W.	-
Ramie	-	-	-	Large regular lumen, highly thickened C.W. are unlike any other fibre

Note C.W. means cell wall

Table 3 (cont'd)
Lumen and Cell Wall Characteristics

Species	Authors			
	Mathews (1952)	Florian (1990)	Koch (1963)	Mukhopadhyay, Bandyopadhyay & Mukhopadhyay (1985)
Flax	Lumen can be filled with yellowish residue	Thick C.W. and small lumen	Lumen narrow and sharply defined	-
Hemp	Lumen broad, but becomes like line near end of fibre, seldom any contents, sometimes hard to see (less transparent than flax)	Lumen broad and C.W. thick but thinner than flax	Lumen usually broad and frequently indistinct	-
Jute	Lumen sometimes wider than C.W., oval or round (transverse), can be thin line (longitudinal), has constrictions and irregular thickness in C.W.	Thick C.W., fibre cell can be variable width along length but C.W. is constant	Width of lumen varies greatly within single fibre	1°C.W.-fibrils generally crisscross arrangement, 2°C.W. straight, almost parallel layers, some helically twisted fibrils(Z) mostly under straight fibrils and present in upper portion of 2°C.W. only
Ramie	Lumen may contain material, large, open, may be indistinct, has heavy C.W.	Lumen usually filled with contents	Has points of twisting	-

Note C.W. means cell wall.

Table 3 (cont'd)
Lumen and Cell Wall Characteristics

Species	Authors			
	Rice (1963)	Sen & De (1949)	Strellis & Kennedy (1967)	The Textile Institute (1975)
Flax	Lumen larger and more easily seen than in hemp	-	Lumen of uniform width, very narrow, almost line-like, often indistinct, can be closed, walls thick	Very thick walls, very small lumen, C.W. spiral fibrillar structure, 1°C.W. has S-twist
Hemp	Lumen smaller and less easily seen than in flax	-	C.W. thick, lumen wider than flax, always wide open and continuous	1°C.W.fibrillae run in a Z twist
Jute	-	Fibrillar arrangement-highly oriented structure of 2°C.W. enveloped in sheath of flat Z twisted fibrillar wrapping of 1°C.W.	Thick C.W., smooth, lumen varying in width throughout, round or oval(transverse), broad, well defined, but sometimes closes up or is missing for short distance	Thick walls, lumen can broaden causing C.W. to be thin in areas, circular to elliptical lumen
Ramie	-	-	C.W. thick, lumen not wide, but well defined, often has granular material	-

Note C.W. means cell wall.

Table 3 (cont'd)
Lumen and Cell Wall Characteristics

Species	Authors	
	Heyn (1954)	Batra (1985)
Flax	Lumen may be discontinuous and closed at some places	Lumen is narrow line completely filled with protoplasm
Hemp	Lumen is always continuous	
Jute	Irregular width of lumen, sometimes closing up	
Ramie	Thin walls, wide lumen, longitudinal fissures or cracks traverse wall from outside toward lumen, lumen contains remnants of protoplasm	Thin, well defined lumen of irregular shape, C.W. has tendency to develop radial cracks

Note C.W. means cell wall.

Table 4
Dislocations - Nodes

Species	Authors			
	Anderson (1927)	Catling & Grayson (1982)	Garner (1966)	Heyn (1954)
Flax	Present, believes they are due to mechanical injury	Frequent and conspicuous	Present	Markings may be single or double making cross-marking and local swelling of fibre (node), probably result from mechanical influences
Hemp	-	Frequent and pronounced	Present	As in flax
Jute	-	Occur regularly but not as numerous as other bast fibres	Absent	Absent
Ramie	-	Frequent	-	Dislocations faint, less pronounced than in flax

Table 4 (cont'd)
Dislocations - Nodes

Species	Authors			
	Mathews (1952)	Batra (1985)	Mukhopadhyay, Bandyopadhyay & Mukhopadhyay (1985)	Rice (1963)
Flax	-	Nodes plentiful, in form of X, on extension of fibre they disappear; on relaxation, they reappear	-	Present
Hemp	Frequent	Frequent	-	Present, but not so pronounced as in flax
Jute	-		Nodes present in 1° layer only, with odd exception	-
Ramie	Frequent		-	-

Table 4 (cont'd)
Dislocations - Nodes

Species	Authors		
	Strellis & Kennedy (1967)	Florian (1990)	Koch (1963)
Flax	Clear, transverse dislocations, in shape of I, X, or V bulges, more conspicuous than in other bast fibres	Has nodes along length of fibre	Transverse and oblique cracks (dislocations) and nodular swellings
Hemp	-	-	As with flax
Jute	Numerous	Has nodes along length of fibre	No dislocations
Ramie	-	Has nodes along length of fibre	Has dislocations

Table 5
Cell Ends

Species	Authors			
	Catling & Grayson (1982)	Garner (1966)	Kundu & Sen (1960)	Batra (1985)
Flax	Many tapering and rounded or have thread-like tips, a few bifurcated	Pointed	-	May be forked
Hemp	Bluntly pointed tips most usual, can have round, tapering and rounded, pointed and tapering, pointed ends, also bifurcated, unequally bifurcated, scimitar-like, spatulate	Blunt	-	Ends thick walled and blunt
Jute	Every type present in large quantity, can have bud-like projections some distance from tip	Blunt	-	-
Ramie	More often bifurcated than hemp, rounded most common, some tapering and rounded, also bluntly pointed, spatulate, unequally bifurcated and scimitar-like	-	Attenuated ends, tips rounded and often dovetailed with neighbouring fibres, basal tip wider than apical tip	-

Table 5 (cont'd)
Cell Ends

Species	Authors			
	Mathews (1952)	Strellis & Kennedy (1967)	The Textile Institute (1975)	Koch (1963)
Flax	Never has forked ends as does hemp, sharp points	Pointed, tapering gradually, more slender and pointed than hemp	-	Generally sharply pointed, occasionally rounded
Hemp	Blunt, thick-walled, occasional lateral branches, no sharp points	More rounded than flax, can be blunt, thick-walled and occasionally have lateral branches	-	Rounded and sometimes split
Jute	Near cell ends lumen broadens out causing C.W. to become very thin	Ends slender and pointed	Pointed tips	Rounded, more rarely split
Ramie	Thick-walled, round pointed lumen reduced to line	Cells taper to narrow, rounded end	-	Distinctly rounded

Note C.W.means cell wall.

Table 6
Presence of Lignin

Species	Authors			
	Florian (1990)	Menzi & Bigler (1957)	Strellis & Kennedy (1967)	Rice (1963)
Flax	Lower lignin content than jute	-	-	-
Hemp	Lower content than jute, more than flax	-	-	-
Jute	Higher than flax and hemp	High lignin content	Fibres have surrounding lignin layer	High Lignin content
Ramie	Almost pure cellulose	-	-	-

The Processing of Flax, Hemp, Jute and Ramie

As has been stated, ramie does not go through the retting process as do flax, hemp and jute. In order to free the fibre from extraneous plant material ramie must be decorticated. Decortication achieves the same results as retting. A description of this process is given in a later section. A description of the processing of flax will first be given because more complete information is available for this fibre. The treatment of hemp and jute is similar to that of flax. For further details on retting and other operations such as bleaching and dyeing, carried out on hemp and jute, reference should be made to Cook (1984), Mathews (1952), and The hemp plant (1962). An extremely detailed account of Belgian flax retteries, with particular attention to those on the river Lys is given by Dujardin (1948).

Flax Processing

The following description, of flax processing, is based on material from *The Weaver's Journal* (Chase, 1982) except where noted. A simple understanding of the procedure is helpful as the same principals are incorporated in hemp and jute processing. The scale of operations for hemp and jute, however, is different because of larger plants and larger crops (The hemp plant, 1962).

When flax is harvested, it is pulled up by the root. Through every step of the process, each stem is kept parallel to the other and the bundles formed are kept even (Chase, 1982).

Before retting can take place, the stems must be rippled with a comb-like device which removes the seed heads. Retting is the most difficult stage to accomplish well. Its purpose is to loosen the individual fibre bundles from surrounding tissue. A partial decomposition occurs which must be suspended before the fibre bundles are broken down completely into individual single-celled fibres unless desired. The pectins holding the fibre cells together are slightly different from those holding the fibre bundles to the pith and cuticle. They dissolve more slowly and with greater difficulty (Chase, 1982).

The retting process itself takes place under very slow moving water (where the action is essentially bacterial in nature) or in the open field by means of dew (where the

action is fungal). Temperature is an important factor in both methods (Chase, 1982). Basu (1980a) states that "the phenomenon of retting is based on the activity of pectinolytic, hemicellulolytic, cellulolytic, proteolytic, sulphur-reducing, bacteria ammonifying and nitrifying organisms, denitrificants, and different types of fungi" (p. 109). He goes on to say that "hemicelluloses, pectins, reducing sugars, tannins and proteins are the principal substances removed from the plants during retting" (p. 109). As retting time increases, so does the amount of hemicelluloses removed (Islam, Bhuiyan & Islam, 1978). According to Sharma (1986), in mature plant tissue, the middle lamella is often converted to calcium pectate and thus cells become firmly cemented together making retting more difficult.

Breaking or braking is the next step. The flax is laid across the wooden jaws of the brake and a wooden handle is brought down hard against it in a chopping motion over the whole length of the straw. The pith and cuticle are broken and removed from the in-tact fibres. Scutching removes the last of the extraneous material (Chase, 1982).

Finally, hackling is done with extremely sharp steel pins set into a block of wood. The hank of flax is flipped over the top of the hackle and drawn across it. Moving from the ends gradually to the centre, hackling cleans and aligns the fibre bundles as well as splitting them into finer and finer fibre strands called line. The shorter pieces remaining in the block are called tow. Finally, the fibres are spun into yarn (Chase, 1982) and if required, bleached to white. Bleaching also helps remove any residual lignin and furthers the process of breaking down fibre bundles into smaller groupings. Sometimes they are broken down to individual fibres or ultimates (Batra, 1985). This is usually so with soft linen that is used in apparel.

Ramie Processing

Ramie does not go through the retting process in order to be made commercially useable. The removal of gummy polysaccharides must be facilitated (Ray,1975). This is a difficult process as the gum is highly resistant to bacteria and fungi. According to Kirby (1963), successful degumming experiments have been done in France and Japan

using several strains of anaerobic bacteria. No literature has been encountered which states whether bacterial degumming is in common use today.

Initially, after the ramie canes are cut down, they are topped, stripped of leaves and placed in slow-moving water. A fermentation process takes place which softens the outer bark and releases the fibres from the cane. They can be partially released from each other at this stage, as well (Mathews, 1952).

The fibres are next stripped from the canes while still wet and scraped by hand with tools made of wood, shells, bamboo, bronze or iron. Today, on a large scale, this process is performed by machine (Cook, 1984; Mathews, 1952). The now fluid outer bark and some of the gum is removed this way. The resulting ribbons of fibres, or China grass, are hung to dry. They still contain 30 to 35% gum, but Kirby (1963) claims the figure is 20 to 30% and that the most difficult gum to remove is the last 1 to 2%. Until recently, ramie was exported in this state (Mathews, 1952).

The process thus far is called decortication. Next comes the degumming process. According to Mathews (1952), originally the China grass was washed repeatedly until a large amount of the gum was removed. It was then sun dried. In China, summer clothing has been made from unspun ramie that is only partially degummed (Kirby, 1963). Now, degumming is done chemically. Most methods use alkalis mixed with soap or a similar acting colloid (Mathews, 1936). These particular alkalis break down the pectins in the ramie ribbons, but do not attack the cellulose (Kirby,1963).

As already stated, another method of degumming uses a solution impregnated with bacteria, although it is unclear whether this process is still used (Dujardin, 1948; Mathews, 1952). Gums and waxes are attacked, but cellulose is not.

One Japanese degumming process requires that the fibre ribbons be boiled with lye for from three to four hours. About 35 per cent of the gums in it are thus removed. Next the fibre ribbons are put through a crushing and washing machine and then sprayed with water which both separates the fibres and removes adhering matter. It is then beaten by hammer mills to loosen and soften the fibres. Finally, it is washed by hand, oiled and air-dried (Kirby, 1963).

A Chinese degumming method involves many steps such as soaking in potassium alum solution or aluminum sulphate, washing, boiling in caustic soda solution, washing again and bleaching. It is then boiled, washed, bleached and washed again (Kirby, 1963).

For a more detailed account of ramie processing, referral to Cook (1984) and Kirby (1963) is recommended. Kirby (1963) and Batra (1985) state that the paucity of details on ramie degumming exists because most firms involved in degumming have their own processes and these are kept secret. A great deal of water is used in all degumming (O'Shea, 1989).

Degradation of Cellulose

Introduction

According to Potts (1978), some of the contributing factors to the degradation of cellulose are the cumulative effects of sunlight, especially the ultraviolet component, heat, oxygen, water, chemical pollution, microorganisms such as fungi, bacteria and actinomycetes, insects, and animals, plus the effects of wind and rain. Put another way, "the speed with which a compound breaks down depends [on its] stability in the presence of light, humidity, ...gases in the atmosphere, heat, acids and alkalis" (Landi, 1985, p. 14). These factors exert an influence individually as well as through complicated interactions, including, both mechanical and biological attack (Basu & Ghose, 1962; Landi, 1985).

When textile artifacts survive for long periods of time, they have been preserved under very specific conditions. Extreme desiccation, which is one such condition, does not allow for the existence of most destructive microorganisms, while immersion in liquid prevents decay by aerobic bacteria (King, 1974; Goodyear, 1971). Charring is the controlled oxidation of a fibre such that the macro-structure of the fibre itself is not disrupted. It is a very slow form of combustion where the speed of reaction is controlled by the limited supply of oxygen (Cooke, 1990; Goodyear, 1971).

Intimate contact with copper or bronze can produce pseudomorphs which are records of textiles or other objects. Mineral crystals take on the form of another material.

Mineral deposits, for example, can show the actual fibre and weave of a textile that no longer exists. They can take on the form of that textile (Vollmer, 1974).

There are two separate periods of degradation in the life of a textile; the time during active use and then the period after use when it can be classified as an artifact. Frequently, the textile will be in an archaeological context longer than it has been in a cultural one. Interaction with the environment can be quite different in each period (Jakes & Sibley, 1983).

The beginnings of chemical deterioration in cellulose material occur first at the margins of crystalline areas and in amorphous areas between crystalline micelles. It progresses through to regions between aggregations of the cellulose polymer in the microfibril and between microfibrils (Kronkright, 1990). The initial degradation of amorphous areas is faster than the later stages where the crystalline areas are attacked (Peters,1963). The cellulose polymer degrades through chemical reactions involving the breaking of bonds in the main chain of the molecule (Schnabel,1981).

The chemical degradation of cellulose and related materials is extremely complex. It will be introduced in this paper only briefly. Kronkright (1990), and Peters (1963), each discuss it in considerable detail with Kronkright relating it directly to potential processes that bast fibres can undergo in manufacturing.

Chemical Degradation of Cellulose

Hydrolytic Degradation of Cellulose

Hydrolytic degradation of cellulose as shown in Figure 4 is the acid catalyzed decomposition of the molecule with the addition of water (Jakes & Sibley, 1983). Products may have a wide range of degrees of polymerization and are referred to as hydrocelluloses (Peters, 1963). Either acidic compounds or oxidation provide hydrogen ions which in combination with water break the 1,4 - carbon - oxygen bond (Kronkright, 1990). Degree of polymerization is lowered and reducing and non-reducing groups are formed. The chain length decreases and the chains break at random points. The

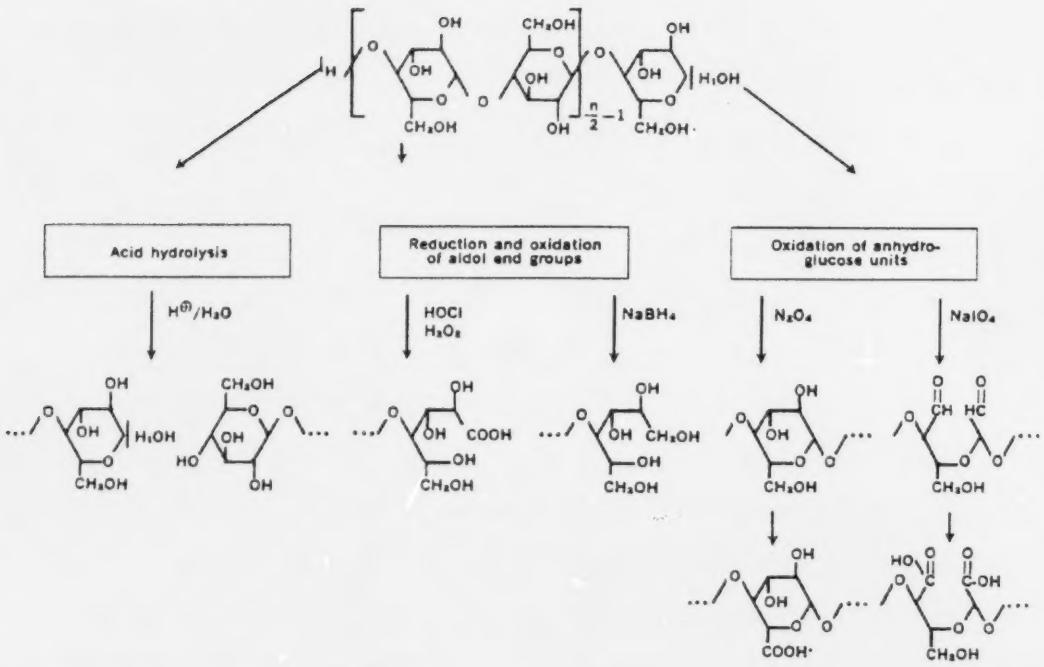


Figure 4 Reactions of cellulose.

Note. Adapted from "Chemical Reactions with Cellulose Fibers" by H. Hollinger, 1972, *Journal of the American Association of Textile Chemists and Colorists, 49, November, p.249. Adapted by permission.*

cellulose has a high reducing power if the chain has been broken many times. There are no significant numbers of single molecules of glucose early in the reaction (Peters, 1963).

The reaction occurs at a faster rate with increased temperature, moisture content and lower pH. Both cellulose in the cell wall and hemicelluloses from the middle lamella are subject to decreases in the degree of polymerization by acid hydrolysis according to Kronkright (1990). He succinctly describes the consequences of hydrolysis:

> These shorter polymers are less capable of withstanding loads placed upon them... The solubility of the polysaccharides and their component acids and sugars increases. A loss of moisture regain and amorphous regions occurs along with an increase in crystallinity, resulting in changes in the homogeneity of the cell wall... Carboxyls and carbonyls increase, resulting in colour changes and increased sensitivity to light degradation [and to alkaline degradation]. Acidity of the material also increases, mostly because of the acidic byproducts of hydrolysis (p. 166).

Oxidative Degradation of Cellulose

In the oxidation of cellulose, oxygen is added (see Figure 4). Oxidation can take place in acidic, neutral or alkaline environments though it is more likely to occur in an alkaline environment (Jakes & Sibley, 1983; Peters, 1963). At least four points on the glucose molecule are vulnerable to attack by oxidizing agents producing a variety of products known as oxycelluloses. Both acidic and reducing types of oxycellulose exist (Trotman, 1984; Peters, 1963). It seems that most oxidizing agents attack cellulose in a non-specific manner, while a few such as nitrogen dioxide are specific in the site that they oxidize (Peters, 1963).

Aldehyde groups can be oxidized to carboxyls as one possible reaction (Peters, 1963). The resulting products are "copolymers of glucose and a variety of oxidized glucose structures. The oxycellulose produced by alkaline oxidation is called a 'non-reducing' oxycellulose because it cannot be oxidized further and cannot reduce another compound" (Jakes & Sibley, 1983, p.34). It can have a large quantity of acid groups (Peters, 1963).

In acidic or neutral environments the oxycellulose produced is a reducing one, containing some aldehyde and ketone groups (Jakes & Sibley, 1983). When they are oxidized to carboxly groups, they, in turn cause reduction of something else.

Lee, Feller and Bogaard (1985) state that radiation of wavelength longer than 320 nm can facilitate oxidation, probably through photosensitization. Once absorbed by cellulose it can be dissipated throughout the polymer on all levels of organization. When energies are sufficient, breakage of bonds occurs. In the absence of oxygen, cellulose is not very photosensitive. In its presence, however, degradation takes place at a slow rate (Kronkright, 1990).

As with all chemical deterioration, the crystalline areas of a cellulose fibre are the least vulnerable to attack. Certain transition metals can act as catalysts in the oxidation of cellulose while silver, magnesium and organic antioxidants serve as inhibitors (McBurney, 1954).

Thermal Degradation

As with light energy, thermal energy which is absorbed can be dissipated throughout a polymer at all levels of organization. When energies are sufficient to cause bond breakage, a reaction has occurred (Kronkright, 1990). Cellulose can oxidize quickly at temperatures above 140° C in the presence of molecular oxygen. At lower temperatures the same process can occur but does so at a much slower rate. Moisture serves to accelerate the process. At temperatures above 200° C., carbon dioxide, carbon monoxide, water, glucosan, and other hydrocarbons are produced.

Under thermal degradation, cellulose fibres darken and become brittle. Brittleness occurs partly because amorphous areas break down first and the now denser fibres have increased crystallinity. Brittleness is increased by cross-linking and shortening of the cellulose polymer. Finally, the fibres completely break down (Jakes & Sibley, 1983).

The oxidation reactions that take place at higher temperatures affect both cellulose, hemicelluloses and lignin. Hemicelluloses and lignin start to degrade at lower temperatures than does cellulose with lignin being slightly less thermally stable than the hemicelluloses. The degree of polymerization in cellulose is lowered. Physical swelling takes place independent of that which occurs at room temperature and with the addition of moisture. Desiccation and finally shrinkage occurs. Other chemical reactions can also be accelerated in the presence of thermal energy. Bast fibres experience changes in permeability, colour, surface gloss, texture, resistance to abrasion, changes in moisture regain and swelling (Kronkright, 1990).

Microbiological and Fungal Degradation

As in reductive and oxidative degradation, cellulose fibres are attacked microbiologically in amorphous areas first. Organisms can gain entry more easily through amorphous areas, whether from the outside of the fibre cell or through the lumen (Jakes & Sibley, 1983). Blanchette, Nilsson, Daniel and Abad (1988) state that in wood, fungi grow from cell to cell through openings such as pits or by producing bore holes. A requirement of microbial degradation is a relative humidity above 60 per cent (Kronkright, 1990). Each organism has a specific need for certain nutrients, temperature,

pH and amount of oxygen (or lack of it) (Potts, 1978). Bacterial degradation occurs at a slower rate than fungal decay so that if a fibre and the environment will support fungal growth, bacteria will not be able to compete (Blanchette et al, 1988).

Both fungi and bacteria break cellulose down through hydrolytic and enzymatic reactions into its component glucose molecules rendering it both water soluble and digestible. Bacteria require either an aerobic or an anaerobic environment to survive, but fungi can only exist in an aerobic medium (Jakes & Sibley, 1983). Both bacteria and fungi can damage a fibre cell externally and internally through the lumen, but not always at the same time. The site of damage can be determined by the species of microorganism and by the condition of the fibre cell (Basu & Ghose, 1962). Some fungi can destroy the entire secondary cell wall from the lumen where the hyphae are located (Blanchette et al, 1988).

Bacteria and fungi contain enzymes known as carbohydrases that hydrolyze polysaccharides (Schnabel, 1981). One carbohydrase, cellulase, is an enzyme that catalyses the hydrolysis of cellulose thus aiding in breaking the main chain of the molecule. Two types of cellulase exist: endocellulase and exocellulase. Endocellulase attacks the molecule randomly so that the ultimate end products are cellobiose and glucose. Exocellulase involves the non-reducing end of the polymer and catalyses attack at the second and third glucoside ring from the end. In this case, cellobiose and cellotriose are the end products (Schnabel, 1981).

Some environments hinder the activities of fungi and bacteria: a pH of less than five; high salinity; high concentration of heavy metals and low relative humidity. They prefer either well aerated or anaerobic conditions. Alkalinity and moisture also promote the growth of fungi and bacteria (Jakes & Sibley, 1983).

CHAPTER 3 MATERIALS AND METHODS

Materials Tested

Known Specimens

The research undertaken involved the use of both known fibre specimens of flax, hemp, jute and ramie and a series of unknown samples of textile artifacts. The known specimens were gathered from various sources with enough information about their origins to guarantee their fibre content. A description of each known fibre specimen appears in Tables 7 through 10. Many of these specimens consisted of pieces of fabric. During testing and investigation, such specimens were actually considered as two specimens: a warp and a weft. In the tables the fabric specimens, whether yarn or fabric, are listed as one.

Table 7
Description of Flax Fibre Specimens

#	Source	Structure	Degree of Bleaching
1	TestFabrics L - 51[a]	Tabby fabric	Bleached
2	TestFabrics L - 52[a]	Tabby fabric	Bleached
3	TestFabrics L - 53[a]	Tabby fabric	Warp: bleached Weft: unbleached
4	TestFabrics L - 54[a]	Tabby fabric	Half bleached
5	TestFabrics L - 61[a]	Tabby fabric	Bleached
6	Yarn from Glimåkra[b]	Hand woven damask	Half bleached
7	Yarn from Glimåkra[b]	Hand woven Bronson Lace	Bleached
8	Fleece Artist[c]	2 ply Irish linen yarn	Bleached
9	Fleece Artist[c]	2 ply Irish linen yarn	Half bleached
10	Unspun fibre from Handcraft Wools[d]	Single ply yarn	Unbleached

[a]TestFabrics Inc., P.O. Drawer O, 200 Blackford Ave., Middlesex, New Jersey 08846
[b]Glimåkra Looms 'n Yarn, Inc., 1304 Scott Street, Petaluma, California 94952
[c]Fleece Artist, P.O. Box 881, Halifax, Nova Scotia B3J 2V9
[d]Handcraft Wools, Streetsville, Ontario, no longer in business

Table 8
Description of Hemp Fibre Specimens

#	Source	Structure	Degree of Bleaching
1	Market near Zagreb, Yugoslavia	Single ply yarn	Unbleached
2	Market near Zagreb, Yugoslavia	Single ply yarn	Unbleached
3	Market near Zagreb, Yugoslavia	Single ply yarn	Unbleached
4	Market near Zagreb, Yugoslavia	Single ply yarn	Unbleached
5	Yugoslavia	Unspun retted fibres	Unbleached
6	McCrone Research Associates Ltd.[a]	Unspun retted fibres	Unbleached

[a]McCrone Research Associates Ltd., 2820 South Michigan Avenue, Chicago, Illinois 60616

Table 9
Description of Jute Fibre Specimens

#	Source	Structure	Degree of Bleaching
1	Purchased by Mrs. H. Bentley in Edmonton, Alberta, 1968	Tabby fabric	Unbleached
2	Purchased by Mrs. H. Bentley in Edmonton, Alberta, 1968	Tabby fabric	Unbleached
3	Purchased by Mrs. H. Bentley in Edmonton, Alberta, 1968	Tabby fabric	Unbleached
4	Bangladesh	Tabby fabric	Unbleached
5	National Research Council[a]	Tabby fabric	Unbleached
6	McCrone Research Associates Ltd.[b]	Unspun retted fibres	Unbleached
7	Action Furniture Upholstery, Edmonton, Alberta	Tabby fabric	Unbleached
8	Handcraft House[c]	4 ply cordage	Unbleached
9	Romni Wool and Fibres[d]	Unspun retted fibres	Unbleached
10	Society for the Preservation of New England Antiquities[e]	Tabby fabric	Unbleached

[a]National Research Council, Ottawa, no longer has textile section
[b]McCrone Research Associates Ltd., 2820 South Michigan Avenue, Chicago, Illinois 60616
[c]Handcraft House, North Vancouver, British Columbia, no longer in business
[d]Romni Wools and Fibres, 5345 West Boulevard, Vancouver, British Columbia V6M 3W4
[e]Society for the Preservation of New England Antiquities,185 Lyman Street, Waltham, Massachesetts 02154

Table 10
Description of Ramie Fibre Specimens

#	Source	Structure	Degree of Bleaching
1	National Research Council[a]	Single ply yarn	Bleached
2	Irish Linen Shop, Edmonton	Tabby fabric	Bleached
3	McCrone Research Associates Ltd.[b]	Unspun processed fibres	Bleached
4	National research Council[a]	Unspun processed fibres	Bleached
5	Crescent Fabrics, Downsview, Ontario	Single ply yarn	Bleached
6	Woeller, Kitchener, Ontario	Pile of fabric only	Bleached and very slightly dyed
7	Handcraft Wools[c]	2 ply yarn	Bleached
8	Weavers Conference, West Lafayette, Indiana, 1991	Unspun processed fibres	Bleached
9	Weavers Conference, West Lafayette, Indiana, 1991	2 ply yarn	Bleached
10	Weavers Conference, West Lafayette, Indiana, 1991	2 ply yarn	Unbleached

[a]National Research Council, Ottawa, Ontario
[b]McCrone Research Associates Ltd., 2820 South Michigan Avenue, Chicago, Illinois 60616
[c]Handcraft Wools, Streetsville, Ontario, no longer in business

Cleaning the Known Fibres

All of the above specimens were wet cleaned in a 0.2% solution of Shurgain® anionic detergent and tap water at a temperature of approximately 37° C. The specimens were manipulated by hand for several minutes in the wash bath and then rinsed three times in room temperature distilled water. They were allowed to air dry on a flat clean surface.

Unknown Specimens

In order to qualify as subjects for study, the unknown specimens were initially identified microscopically as being bast fibres. A cellulose fibre such as cotton which is immediately identifiable under optical microscopy did not qualify for further

investigation. There was, however, no guarantee that the selected unknowns were soft fibres. There was always the possibility that they could be hard fibres.

Although the unknown specimens were not wet cleaned, one specimen had been previously washed during conservation. Cleaning was not done, as potentially valuable information such as deposits from salt water and hyphae from microorganisms could be lost. Several of the pieces were very degraded and would not have withstood wet cleaning.

Test Methods

Microscopic Analysis

Initial observations of the fibres were made with an optical microscope to determine with certainty that the known specimens were bast fibres and that none of them were blends of more than one type of fibre such as flax and cotton or ramie and a man-made fibre. An Olympus Binocular KH Biological Microscope was used at a power of 100X. Degree of processing, presence or absence of degradation of individual cells and presence of debris were also noted in the known specimens. When the unknown specimens were viewed microscopically, the above observations were made when possible. Additional note was made of any apparent structural characteristics such as nodes and lumina.

Chemical Test Methods

There are many chemical tests in the literature used for the identification of flax, hemp, jute and ramie. Most rely on colour change or swelling effects for results, either with or without the aid of a microscope. In this research, the tests not chosen for in-depth investigation were eliminated for several reasons. Unfortunately, most authors will refer to a particular test to verify a fibre identification without actually stating whether they have performed the test. Some authors use such tests as part of their research, but while giving the results of a test, they will seldom give the method. As well, different procedures for the same tests exist and they may not all give precisely the same results.

Three chemical tests were used on all sets of fibre specimens, both known and unknown. They were chosen because they appeared frequently in the literature, detailed methods were available, reagents were not difficult to obtain, and results from the known specimens were consistent and emphatic. Without exception, however, none of the literature consulted made more than a passing reference to the chemistry involved; neither the interaction of the chemicals used to make up the various test materials nor how these materials reacted with the fibres being tested was discussed.

Phloroglucinol and Hydrochloric Acid

The first reagent applied to the fibres was phloroglucinol [$C_6H_3(OH)_3.2H_2O$] and hydrochloric acid [HCl]. Phloroglucinol acts as a reagent for both hydrochloric acid and lignin and thus will stain lignin magenta (Merck, 1968).

A Pantone® by Letraset™ colour selector was first used to help determine the colour of the samples before testing. The Pantone® colour selector consists of strips of paper with many different hues, tints and shades. Each one has a number. The samples were viewed, along with the Pantone® colours, in a Dianolite™ box under simulated daylight. The Dianolite™ box has a neutral grey interior and uses a fluorescent light source to simulate daylight. The correlated colour temperature is 6500°K with its maximum energy

Table 11
Typical Results obtained by Researchers using Phloroglucinol and Hydrochloric Acid on Flax, Hemp, Jute and Ramie

Species	Authors		
	The Textile Institute (1975)	Koch (1963)	Harris (1954)
Flax	Traces of pink or nothing	Brown with traces of pink (raw flax)	Traces of pink or nothing
Hemp	Traces of pink or nothing	Pink	Traces of pink or nothing
Jute	Magenta	Violet red	Magenta
Ramie	Traces of pink or nothing	-	Traces of pink or nothing

in the blue end of the spectrum (Diano Corporation). Because the Pantone® colour selector has no whites or cream colours, only fibre samples of beiges or browns were given numbers.

Five samples of each specimen were simultaneously immersed for exactly 5 minutes in the phloroglucinol and hydrochloric acid solution mixed according to Harris (1954) and timed with a stop watch. The method can be found in Appendix A-1. Where specimens had a warp and a weft, a total of 10 samples were tested for each specimen.

After immersion, the samples were removed to acid-free blotting paper where they were allowed to drain briefly. They were then placed on a glass slide over white paper and viewed under simulated daylight in the Dianolite™ box. They were first compared to the original specimen and then to the Pantone® colour selector. Where possible, the immersed samples were assigned a Pantone® number.

Next, several fibres from each sample were mounted on glass slides in distilled water. They were immediately observed under the optical microscope at 100X. Colour intensity and distribution of colour were noted.

Table 11 gives the results of three authors for phloroglucinol and hydrochloric acid testing of flax, hemp, jute and ramie. Methods are not given because they are seldom encountered in the literature. The table is presented as a means of comparison with results obtained from the testing in this study.

Zinc Chloroiodide (Herzberg Reagent)

The second reagent applied was zinc chloroiodide (Herzberg reagent). The precise formula for this unstable reagent could not be found. The preliminary colour evaluations done in the phloroglucinol test were not repeated. Five samples of each specimen were immersed for exactly five minutes in the solution mixed according to the Textile Institute (1975). The method can be found in Appendix A-1. It was necessary to submerge them with a glass rod to remove air bubbles. The test solution was replenished after approximately every five submersions because it would exhaust, causing staining to start becoming fainter. Where specimens had a warp and a weft, a total of ten samples were tested for each specimen.

Unlike the phloroglucinol test, Pantone® numbers were not assigned to any of the stained samples. As the colour changes were often very subtle and partly served the purpose of highlighting cellular structures, they were viewed only with the optical microscope under 100X using the zinc chloroiodide solution as mounting fluid. When water was tried as a mounting fluid, the stained fibres quickly faded whereas when the zinc chloroiodide solution was used, colour was maintained, but did not increase or decrease over the viewing time of several minutes.

According to Koch (1963), zinc chloroiodide solutions will break down with time and iodine will precipitate out of the solution, causing the colour changes to be different from those where a fresh solution has been used. It was discovered that the solution would last approximately five days before it would break down. Therefore, this test was administered to all specimens within four days of preparing the reagent.

Table 12
Typical Results obtained by Researchers using Herzberg Reagent on Flax, Hemp, Jute and Ramie

Species	Authors			
	Koch (1963)	Strellis & Kennedy (1967)	Willard (1952)	Rice (1963)
Flax	Bluish violet, impurities yellow	Red with yellow lumen contents	Red	Brownish violet
Hemp	Blue or greenish, lignified portions yellow	Blue or violet with traces of yellow	Blue	True violet or violet with traces of yellow
Jute	Yellowish brown	-	Yellow	-
Ramie	Bluish violet	Brownish purple, blue or red	-	Blue

Table 12 (cont'd)
Typical Results obtained by Researchers using Herzberg Reagent on Flax, Hemp, Jute and Ramie

Species	Authors	
	Mathews (1952)	Heyn (1954)
Flax	Brownish violet	Deep blue violet, dislocations - intensely blue; lumen - yellow
Hemp	True violet	-
Jute	-	Yellowish brown
Ramie	Blue	Violet blue

The results of six authors for zinc chloroiodide testing of flax, hemp, jute and ramie are shown in Table 14. Although detailed methods are not given, with the possible exceptions of Willard (1952) and Mathews (1952), it can be assumed that colour changes were observed under optical microscopy. Table 12 is presented in order that the comparison of results obtained by other researchers may be made with results found in this study.

Cuprammonium Hydroxide (Schweitzer's Reagent)

The third test applied was cuprammonium hydroxide (Schweitzer's reagent). The precise formula for this reagent could not be found. In the final step of the preparation of cuprammonium hydroxide, cupric hydroxide [$Cu(OH)_2$] is dissolved in a small amount of concentrated ammonium hydroxide [NH_4OH]. Once these two substances are mixed, the resulting cuprammonium hydroxide is used within two days as it is not a stable solvent.

Unlike the previous two tests this one induces swelling and dissolution of cellulose. Peters (1963) states that cuprammonium hydroxide is alkaline and causes oxidation of cellulose. Because of the characteristic amounts and distribution patterns of lignin in each of flax, hemp, jute and ramie, the swelling and dissolution patterns are also individual

in each of the four fibres. The method used for this test was from Koch (1963) and can be found in Appendix A-1.

No colour evaluations were required for this test. Five samples of each specimen consisting of only a few fibres each were placed on a glass microscope slide on the viewing stage of an optical microscope. Enough of the solvent was added with a dropper to saturate the samples and then a glass cover slip was placed on top. A power of 100X was used for initial observation of swelling and dissolution and then 400X to observe

Table 13
Typical Results obtained by Researchers using Schweitzer's Reagent on Flax, Hemp, Jute and Ramie

Species	Authors			
	Menzi & Bigler (1957)	Mathews (1952)	Koch (1963)	Hearle & Peters (1963)
Flax	Swells but does not completely dissolve	Swells but does not completely dissolve	Rapid swelling and dissolution; fine corrugated thread of protoplasm generally in interior	-
Hemp	Swells unevenly, then dissolves, leaves fragments of parenchyma	Swells unevenly, then dissolves, leaves fragments of parenchyma	Slow swelling and dissolution; often criss-cross swelling of C.W.	Lignin toward outside of walls so walls swell inward, lumen may become occluded
Jute	-	-	Marked swelling without dissolution; macerated fibres are dissolved	Lignin more evenly distributed than in hemp, swelling causes increase in external dimensions
Ramie	No reaction, swells but does not dissolve	No reaction, swells but does not dissolve	Marked swelling and dissolution	-

Note C.W. means cell wall.

Table 13 (cont'd)
Typical Results obtained by Researchers using Schweitzer's Reagent on Flax, Hemp, Jute and Ramie

Authors	
Heyn (1954) Flax	Heyn (1954) Hemp
Beginning of swelling: Very fine layers can be seen in the C.W. The fibre swells laterally and contracts lengthwise, compressing the protoplasm in the lumen so that it has the appearance of an undulated snakelike thread (this is characteristic of flax and distinguishes it from hemp). Ballooning can happen and finally the whole fibre dissolves leaving crinkled protoplasmic threads.	Beginning of swelling: It is slower than flax with the C.W. layers appearing thicker. The fibre contracts but there is no undulating thread (less protoplasm). A strongly developed middle lamella shows during contraction. It becomes a ruffled band of accordion-like pleats which remain after the cellulose has fully dissolved.

Note C.W. means cell wall.

details. If the rate of the reaction was rapid, the process was repeated until a consistent pattern of swelling and dissolution was established. If complete dissolution had not occurred within fifteen minutes, the samples were left for twenty-four hours and observed again.

Table 13 gives the results of five authors for the microscopic observations done while swelling flax, hemp, jute and ramie in cuprammonium hydroxide. The results among authors vary greatly, suggesting that different concentrations of the reagent were used.

Other Test Methods

The Twist Test

The twist test, according to Hock (1942), was performed on five fibres from each specimen. Two to three fibres, 2.5 to 5.0 centimetres in length were carefully separated from the yarns and one end was glued with rubber cement at right angles to a 1 cm^2 piece of paper. The paper was held while the fibres were immersed in warm water for thirty seconds. The free ends of the fibres were then held with tweezers, allowing the attached paper to hang freely. As the fibres dried, they rotated in a characteristic direction.

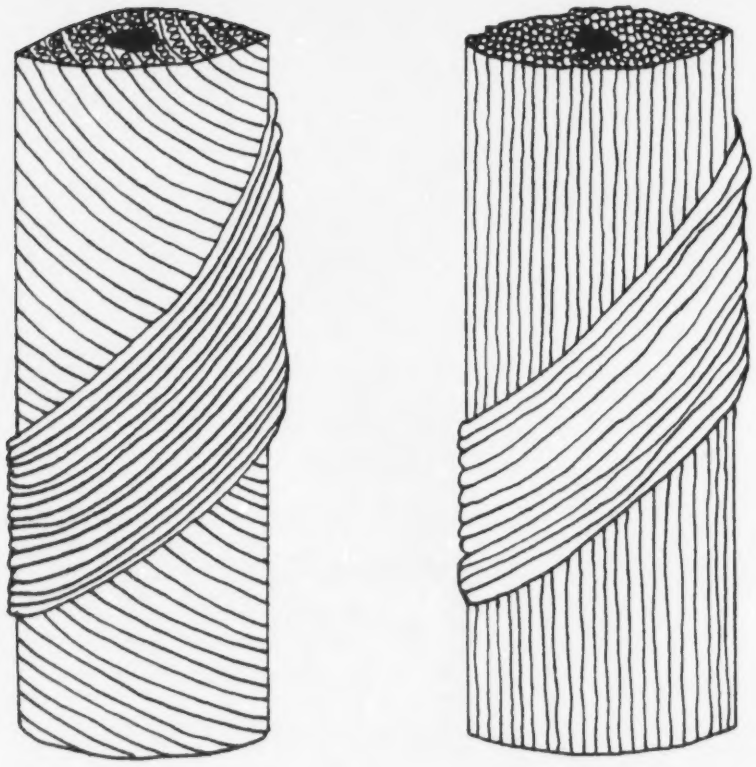


Figure 5. The arrangement of microfibrils in (a) flax and ramie, and (b) hemp and jute.

When looking down the axes of the fibres, flax and ramie should twist in a counterclockwise direction, while hemp and jute, in a clockwise direction.

Hock (1942) explains that the direction of rotation as a wet fibre dries indicates fibrillar orientation, with the various directions of twist in the known fibre cells being due to their inherent structures (Figure 5). The fibres, swollen in water, twist in the same direction as the lay of the microfibrils when the water evaporates. Flax fibre cells consist of many fibrils oriented at an acute angle to the long axis of the cell, with most having an S twist. The outermost layer, however, has a Z twist. Ramie has a similar fibrillar structure to that of flax. With hemp and jute, the outermost layer of fibrils also twists in a Z direction, but most of the underlying fibrils lie nearly parallel to the fibre cell axis.

Because of the neutrality of the parallel fibrils, twist is controlled by the Z direction of the outermost layer only. Sen and De (1949) concur with this.

Fibre Diameter Measurements

Fibre lengths of the specimens could not be measured reliably, particularly those of the unknown specimens as these were sometimes quite small. Even in large samples, ramie cells, for example, can be extremely long; in fact, too long to make microscope slides (Aldaba, 1927). Most often, ramie fibres are unicellular.

While fibre lengths were not measured, fibre diameters were. It was decided that determining the diameters of samples of both the known and unknown specimens would be useful for comparisons with the diameter measurements from the literature in Table 1. If typical and consistent measurements could be found within samples, and to some degree, within species, a means of comparison could possibly be established between the known fibre specimens and the unknowns.

Representative measurements were taken of all the specimens within the groups of known flax, hemp, jute and ramie fibres. Having such measurements of the known fibres allowed fibre diameter measurements of the unknown specimens to be compared with them.

The method used for measuring fibre diameters was compiled partially from Heyn (1954) for the measurement of diameters of natural fibres. First an eyepiece micrometer was calibrated using a stage micrometer. Then a random selection of fibre samples was measured, one species at a time, with no mounting liquid but under a glass coverslip. Water was added drop-wise at the edge of the coverslip. Care was taken not to move the slide. The fibres were measured again and then measured five minutes later to determine if any visible swelling had taken place. This amount of time was more than the time necessary to make fifty fibre diameter measurements. Though it is probable that some swelling would occur under these circumstances, none was perceptible when measurements were taken.

Next, five very small samples were taken from each specimen and mounted in distilled water on a glass slide. Immediately fifty measurements at 315X were taken of

fifty fibres with the eyepiece micrometer mounted on an optical microscope. If the specimen had a warp and a weft, a total of 100 (50 of each) measurements were made.

Measurements were made from the upper left corner of the field of fibres across the top of the field to the upper right corner. The slide was then moved approximately two mm vertically and measuring continued at this level from right to left. Measuring continued in this fashion until fifty fibres had been counted (AATCC, 1991).

After a few preliminary measurements were done on the four species of fibres, it was decided that each species would be measured in a specific way. This was due to the variation found in cell bundle sizes between species.

The flax fibre specimens occurred almost entirely as single cells. Where bundles were present they were small with almost every fibre clearly distinguishable from the others in the bundle. It was therefore decided that measurements would be taken of *single cells* at a point away from the cell end where narrowing had not yet begun.

While the known hemp specimens contained some protruding single fibre cell ends, all cells were incorporated into bundles. The bundles were generally much larger than those of flax and the individual cells in those bundles were not so clearly visible. It was decided to measure *bundles of cells* only. In order to free individual cells from the bundles, it would be necessary to macerate them. Though macerations could be prepared from undegraded specimens, this is not the case when small samples of even minimally degraded artifacts must be measured.

Jute cells, like hemp, were found in bundles. It was more difficult to see individual cells in jute fibres than in hemp. It was, therefore, decided to measure *bundles of cells* only.

Ramie, like flax, was most often found in the single cell state in the known specimens. Where two or three cells were together in a bundle, they were usually easily distinguished from each other. Only *single cell* measurements of ramie were taken at a point away from the cell end where narrowing had not yet begun. An additional reason should be noted for measuring single cells in ramie. Ramie, more than any other bast fibre, is most often confused with flax. It was decided that the most useful comparison

that could be made between the two fibres would be that of both fibres in their single cell states.

Unknown specimen fibre measurements.

Unlike the known specimens, no procedure of measurement could be used consistently with the unknown specimens. If fibres were found to be in a single cell state, they were, of course, measured as single cells. If they were found to be in bundles, they were measured as such. In both cases this was noted along with the measurements. If the specimens were found to have both single cells and cell bundles, both were measured and noted as it is possible that the specimens could consist of a blend of fibres. In a few cases, the specimens were too encrusted and degraded to take fibre measurements.

Statistical analysis of fibre diameters.

Frequencies, means and standard deviations were the only statistical analyses performed on the fibre diameters. Other statistical calculations were not considered of use in comparing the differences in measurements among fibres. It is useful to compare similarities and differences both among the individual species measurements and then between those and the unknown samples. Any recurring similarities, along with similar results in other testing of known species could indicate the species of an unknown sample.

Frequencies of the diameters of each sample were produced in histogram form using the LOTUS Freelance 4.0 program for an IBM compatible personal computer. The histograms are presented in Appendix A-2.

Means were taken of each group of fifty measurements of the four species; one each for flax, hemp, jute and ramie. Then standard deviations were done for the four species as well as confidence intervals. The same statistical calculations were performed on each of the unknown specimens.

SEM Analysis

Scanning electron microscopy is similar to optical microscopy except that instead of a beam of light acting as an illuminating source, a beam of electrons is used. This

method allows for high magnification, precise detail and great depth of field (Bresee, 1984).

Samples consisting of a few fibres were mounted on stubs with double sided adhesive tape. Conductivity through the stub was increased by applying carbon glue to the edge of the sample. The samples were then sputter-coated in a vacuum chamber with 15 nm of 24k gold to provide a conductive surface. They were individually placed in the vacuum chamber in a Cambridge Stereoscan 250 scanning electron microscope where they were viewed and photographed.

Analysis was done on the crystals present on some of the fibres with Energy Dispersive X-Ray Analysis (EDXA). Elements present in sufficient quantities and with molecular weights higher than sodium could be identified.

Photographs of flax, hemp, jute and ramie were taken at different magnifications. Before final photographs were selected, many fibres were first viewed to be sure that those photographed were typical. Each plate of the known fibres has three photographs. The first two are general; the third of each species shows some typical detail.

Included in the SEM photographs are six selected from the unknown specimens. They were chosen because they show the degree and varieties of encrustation and degradation encountered in the unknown specimens.

CHAPTER 4 RESULTS AND DISCUSSION

Microscopic Observation of the Known Specimens

After making initial microscopic observations to determine with certainty that the known specimens were bast fibres and that none of them were blends of more than one type of fibre such as flax and cotton or ramie and a man made fibre, additional observations under 100X optical microscopy were undertaken. An Olympus Binocular KH Biological Microscope was used. Degree of processing, presence or absence of degradation of individual cells and presence of debris were determined. The following Tables 14 through 17 give these observations. More detailed observations which were taken after staining or swelling are presented later on in this chapter.

Microscopic Observation of the Unknown Specimens

Microscopic observations of the unknown fibres were done to be sure that they were soft or hard fibres. The microscopic observations of the unknown fibres take into account more detailed observations than the microscopic observations of the known specimens. As it is more difficult to be consistently precise about observing processing, degree of degradation and number of cells per bundle in the unknown specimens, instead, the most obvious characteristics were reported. The above categories were, nonetheless, noted where possible. Table 18 gives the microscopic observations of the unknown fibre specimens.

Table 14
Microscopic Observation of Flax Fibre Cells in Known Specimens (100X)

#	General Observations	Degree of Processing and Degradation	# of Fibre Cells per Bundle
1 Warp	Clean, very little debris	Processed almost entirely to single cell stage, does not appear degraded or damaged	Most cells found singly, rarely in bundles of 2 or 3
Weft	Same as warp	Same as warp	Same as warp
2 Warp	Same as #1	Same as #1	Same as #1
Weft	Same as #1	Same as #1	Same as #1
3 Warp	Same as #1 but with more debris	Same as #1	Same as #1
Weft	Same as warp	Not as processed as #1 and does not appear degraded or damaged	Has bundles with many different cell numbers/bundle
4 Warp	Same as #1 but with more debris	Processed almost as much as #1, does not appear degraded or damaged	Many single fibre cells and some with many cells/bundle
Weft	Same as #1	Same as #1	Same as #1
5 Warp	Same as #1	Same as #1	Same as #1
Weft	Same as warp	Same as warp	Same as warp
6 Warp	Has a small amount of debris	Slightly less processed than #1, does not appear degraded or damaged	Has a few more bundles of 2 or 3 than #1
Weft	Same as warp	Same as warp	Same as warp
7 Warp	Has a small amount of debris	Same as #1	Almost entirely single cells
Weft	Same as warp	Not quite as processed as warp, does not appear degraded or damaged	A few less single cells than warp
8 Yarn only	Same as #1	Same as #1	Same as #1
9 Yarn only	Clean, very little debris	Not quite as processed as #1, does not appear degraded or damaged	Same as #3 Weft
10 Yarn only	Large amount of debris	Unbleached, not as processed as #1, does not appear damaged or degraded	Same as #3 Weft, but cells are larger scale (more mature)

Table 15
Microscopic Observation of Hemp Fibre Cells in Known Specimens (100X)

#	General Observations	Degree of processing and Degradation	# of Fibre Cells per Bundle
1 Yarn only	Very clean	Processed to bundles with several cells, does not appear degraded	Bundles are 3 or more fibre cells
2 Yarn only	Large amount of debris	Not as processed as #1, does not appear degraded	Almost all have many cells/bundle
3 Yarn only	Very clean	Same as #1	Same as #1
4 Yarn only	More debris than #1 and less than #2	Slightly less processed than #1	Some single fibre cells but most have many cells/bundle
5 Fibre only	A little debris, obvious brown contents in lumen	May only be retted, does not appear degraded	Many cells/bundle
6 Fibre only	A little more debris than #5	Same as #5	Same as #5

Table 16
Microscopic Observation of Jute Fibre Cells in Known Specimens (100X)

#		General Observations	Degree of Processing and Degradation	# of Fibre Cells per Bundle
1	Warp	Clean, very little debris, lustrous	Unbleached but has been processed to degree that produces typical burlap cloth, does not appear degraded	Large number of fibre cells per bundle
	Weft	Same as warp	Same as warp	Same as warp
2	Warp	Same as #1, but more lustrous	Same as #1, but possibly a bit more processed	Bundles are slightly smaller than #1
	Weft	Same as warp	Same as warp	Same as warp
3	Warp	Same as #2	Same as #2	Same as #2
	Weft	Same as #2	Same as #2	Same as #2
4	Warp	Same as #1	Same as #1	Same as #1, but with a few bundles having few cells/bundle
	Weft	Same as #1	Same as #1	Same as warp
5	Warp	Same as #1, but with no lustre	Same as #1	Has bundles with few cells/bundle and very many cells/bundle
	Weft	Same as warp	Same as #1	Same as warp
6	Fibre only	Same as #1	Same as #1	Same as #1
7	Warp	Same as #1	Same as #2	Same as #2
	Weft	Same as #1	Same as #2	Same as #2
8	Yarn only	Same as #1	Same as #1	Same as #4
9	Fibre only	Has some debris and is lustrous	Same as #1	Has bundles with few cells/bundle and many cells/bundle
10	Warp	Same as #1	Same as #1	Same as #1
	Weft	Same as #1	Same as #1	Same as #1

Table 17
Microscopic Observation of Ramie Fibre Cells in Known Specimens (100X)

#		General Observations	Degree of Processing and Degradation	# of Fibre Cells per Bundle
1	Yarn only	Has some debris and possibly gum	Processed almost to single cell stage, but possibly has some gum residue, does not appear degraded	Mostly single fibre cells
2	Warp Weft	Same as #1 Same as #1	Same as #1 Same as #1	Same as #1 Same as #1
3	Fibre only	Very clean	Processed to single cell stage, does not appear degraded	All single fibre cells
4	Fibre only	A little debris and possibly a little gum	Same as #3, but with a little gum	Same as #3
5	Weft only	Same as #4	Same as #4	Same as #3
6	Pile only	Same as #3	Same as #3	Same as #3
7	Yarn only	Same as #4	Same as #4	Same as #3
8	Fibre only	Same as #3	Same as #4	Same as #3
9	Yarn only	Same as #4	Same as #4	Same as #3
10	Yarn only	Same as #3	Processed almost to single cell stage, no gum residue, does not appear to be degraded	Same as #1

Table 18
Microscopic Observation of Unknown Fibre Specimens (100X)

# and Description	Observations
1 Napkin from Mrs. H. Bentley, approx. 1900	Consists almost entirely of single fibre cells with many cell diameters. Has I,X and V shaped nodes as well as swollen nodes. Degradation Has many broken and very frayed cells, many with hair-like strands coming away from C.W. Many have Z direction splits in C.W., some are S direction while a few are | axis of cell.
2 Unknown cordage from Mrs. H. Bentley	Has many bundle sizes and fibre cell sizes, some single cells and small bundles, nodes of all types and narrow lumina. Very large bundles have tapering cell ends and round-cornered oblong air bubbles (possibly inside lumina), and a few nodes. Sample appears to be a mix of 2 distinct fibre types. Degradation Has very little debris; does not appear degraded.
3 Sail from Bishop Museum, Honolulu	Degradation Has a lot of debris and is highly degraded. Has many single cells (due to degradation of middle lamella?) broken at nodes. Nodes generally more degraded than rest of cell. Some lumina are very broad (degraded from within?). There are many large bundles (encrusted together?) where cells are indistinguishable.
4 Canvas from CCI[a]	Has many single fibre cells and many large bundles (some encrusted together?). Only nodes of swollen type are visible. Degradation Fibres have surface encrustations and a few splits in C.W. in a very sharp Z direction.
5 Marine fabric from CCI[a]	Has many single fibre cells and a few bundles with many different cell diameters, many nodes of all types and narrow lumina. Degradation Some bundles are encrusted together. Has many cell breaks at nodes.
6 Fabric from pillow slip, J. Marshall	Consists of almost all single fibre cells with many cell diameters, nodes of all types and many narrow lumina. Degradation Does not appear degraded.
7 Fibres from Egyptian fabric, 1450 B.C., ROM[b]	Has many single fibre cells of various diameters, many very small bundles, narrow lumina and all types of nodes. Degradation Some cells are encrusted together, many are very broken with breaks at some nodes.
8 Fibres from child's Coptic tunic (before washing), ROM[b]	Has many single fibre cells, some small bundles (some encrusted together?), many different cell diameters and a few narrow lumina. Has nodes of all types, particularly swollen and swollen with bends. Degradation Has surface encrustations, but very little degradation.
9 Same as #8, (after washing), ROM[b]	Same as #8
10 Fibres from ROM[b]	Has multi-cell fibres with extraneous tissue and some swollen nodes. Has at least 1 interrupted lumen that becomes quite wide in spots, round-cornered oblong air bubbles. Degradation Does not appear degraded.

Table 18 (cont'd)
Microscopic Observation of Unknown Fibre Specimens (100X)

# and Description	Observations
11 Fabric from conservation lab, U of A	Has many single fibre cells, a few small bundles, various cell diameters, some long tapering cell ends, many nodes of all types and some faint narrow lumina. Degradation Does not appear degraded.
12 Fibres from ROM[b]	Has fine and coarse fibres. Fine Has all single fibre cells or very small bundles, many diameters, many nodes of all types and very narrow discontinuous lumina throughout sample. Coarse Has very large fibre cell bundles, many swollen nodes and only a few lumina visible with varying width throughout cell. Degradation Does not appear degraded.
13 Torrington Pant fabric, Franklin Expedition, #84.8	Has many single fibre cells, a few small bundles, many different diameters, narrow lumina and all types of nodes. Degradation Does not appear degraded.
14 Canvas from Franklin Collection, #82.52	Has mostly small fibre cell bundles. Degradation Has so much encrustation and degradation of fibres that it is not possible to see fibre characteristics.
15 Canvas from Franklin Collection, #82.3	Has single fibre cells, very small bundles, large bundles (encrusted together?), nodes of all types, various diameters and narrow lumina visible in single cells. Degradation Has large amount of debris and encrustations.
16 Cordage from Franklin Collection, #82.62	Degradation Some single fibre cells and small bundles come away from masses which are heavily encrusted and stuck together. Highly degraded cells break up easily. It is not possible to determine cell characteristics but there are a few swollen nodes.
17 Cordage from Franklin Collection, #82.24	Similar to #16 but has nodes of all types on a few single fibre cells. Has narrow and broad lumina (degraded from within?). Degradation Same as #16 but not quite as extreme.
18 Cordage from Franklin Collection, #82.42	Similar to #16 but has no fibre cell characteristics. Degradation Completely encrusted or very degraded.
19 Canvas from Franklin Collection, #82.7	Similar to #17 but can see only a few swollen nodes.
20 Cordage from Franklin Collection, #82.27	Similar to #17 but can see only a few narrow lumina.

[a]Canadian Conservation Institute, Ottawa, Ontario
[b]Royal Ontario Museum, Toronto, Ontario
Note C.W. means cell wall.

Testing of the Known Specimens

The results of the observations with the optical microscope, chemical tests, the twist test, the fibre diameter measurements and the SEM will all be given for the known fibre specimens of flax, hemp, jute and ramie. Once these have been presented, the results for each unknown fibre specimen will be discussed individually. Where possible, tentative or conclusive fibre identification will be given along with the data.

Chemical Tests

The three chemical tests, the phloroglucinol and hydrochloric acid test, Herzberg reagent test and Schweitzer's reagent test were administered to all the fibre samples in the manner described in Chapter 3. The known flax, hemp, jute and ramie fibres were tested first; then the unknown specimens were tested. Results of the unknown tests were compared with those of the known specimens.

Phloroglucinol and Hydrochloric Acid

The phloroglucinol and hydrochloric acid test is for lignin. Tables 19 to 22 give the results of the phloroglucinol and hydrochloric acid testing done on the flax, hemp, jute and ramie fibres. The Pantone® by Latraset™ colour selector was used in the Dianolite™ box under simulated daylight to evaluate colour change. Where a colour change is noted but no Pantone® number is given, either the colour area is too small or the tint is too pale to be recorded by the Pantone® method.

When the known flax specimens were tested with phloroglucinol and hydrochloric acid, the specimens that were originally white did not change colour (see Table 19). Their original whiteness is due to processing which removes extraneous tissue and possibly due to bleaching. Other specimens, however, that were off white or brown showed colour changes from very slight pink to magenta streaks and reddish brown indicating the presence of varying amounts of lignin.

In consulting Table 11 and Table 19, it is possible to conclude that the darker, less processed fibres which changed colour in this test, contain small amounts of lignin. It

Table 19
Phloroglucinol and Hydrochloric Acid Test of Flax Fibre in Known Specimens

#		Original Colour	Colour Change	Colour Change at 100X
1	Warp	White	No colour change	No colour observable
	Weft	White	No colour change	No colour observable
2	Warp	White	No colour change	No colour observable
	Weft	White	No colour change	No colour observable
3	Warp	White	No colour change	No colour observable
	Weft	Brown, Pantone® 467U, with lighter and darker flecks	Reddish brown, Pantone® 484U	No colour observable in some areas, some areas are pink; others brilliant magenta
4	Warp	Off white	No colour change, but 1 small magenta streak	No colour observable except magenta in streak
	Weft	Off white	Same as warp	Same as warp
5	Warp	White	No colour change	No colour observable
	Weft	White	No colour change	No colour observable
6	Warp	Off white	Very slightly pink	No colour observable
	Weft	Off white	Same as warp	No colour observable
7	Warp	White	No colour change	No colour observable
	Weft	White	No colour change	No colour observable
8	Yarn only	White	No colour change	No colour observable
9	Yarn only	Off white	No colour change	No colour observable
10	Yarn only	Light brown, Pantone® 467U	brown, Pantone® 1675U with some areas pink and some magenta streaks	Single fibres show no colour, bundles either have ares of pink or deep magenta

can be seen from these tables that it is the specimens with the most debris and the largest fibre cell bundles that produce colour changes.

While lignin is not necessarily always present in the secondary cell wall or the middle lamella between fibre cells in bundles, it is in these areas that it occurs. As Kawahara (1989) states, it is in its purest form in the middle lamella, but found most abundantly in the secondary cell wall in all lignified cells.

None of the hemp specimens were originally white as they were with flax. It is apparent from Table 15 that the hemp specimens most often occurred in cell bundles and

Table 20
Phloroglucinol and Hydrochloric Acid Test of Hemp Fibre in Known Specimens

#	Original Colour	Colour Change	Colour Change at 100X
1 Yarn only	Beige	Pale orange, Pantone® 156U with pinker overtones	Evenly distributed light pink
2 Yarn only	Beige	Light yellowish dirty pink, Pantone® 488U with darker and more yellow flecks	Overall pink with some magenta areas
3 Yarn only	Beige	Same as #2, but a bit darker,Pantone® 486U with magenta streaks	Overall very pale pink with some intense pink which covers entire bundles
4 Yarn only	Beige	Same as #3	Overall very pale pink with some intense magenta areas
5 Fibre only	Dark beige to light brown, Pantone® 466U	From beige through pink to deep magenta, too much variation for Pantone®	Overall barely pink, a few small areas of brownish red
6 Fibre only	Same as #5	Same as #5, but no deep magenta	Has areas from pale pink to deep pink

were probably not as processed as were most of the flax specimens. The staining, from pale pink to magenta indicates a higher percentage of lignin generally than that found in the flax specimens (see Table 20). Neither Koch (1963), Harris (1954) nor the Textile Institute (1975) make any great distinctions between flax and hemp in terms of colour change with the phloroglucinol and hydrochloric acid reagent. Clearly, this is because in both cases, the amount of lignin can vary depending on the degree of processing of both fibres. No hemp samples remained unstained in this study although it may be possible to have no colour change as Koch (1963) and Harris (1954) report if testing is done on macerations.

Like hemp, none of the jute specimens was originally white. As indicated in Table 16, jute fibre cell bundles are large. Their cell walls are highly lignified as is the middle lamella between cells (Strellis & Kennedy, 1967), thus it is not unexpected that the fibre samples in this study all had magenta staining (see Table 21). This is consistent with the literature.

The ramie specimens originally were all white or off-white. Like flax, they occurred mostly as single fibre cells. Without exception, there was no colour change in the fibres after testing indicating the absence of lignin (see Table 22). This is consistent with the literature as ramie has the highest cellulose content of any vegetable fibre (Basu, 1981) and only 0.6% lignin (Batra, 1985).

Table 21
Phloroglucinol and Hydrochloric Acid Test on Jute Fibre in Known Specimens

#		Original Colour	Colour Change	Colour Change at 100X
1	Warp	Light brown, Pantone® 466U	Deep magenta, Pantone® 240U but more intense	Each fibre bundle uniformly stained from pale pink to magenta with many magenta
	Weft	Same as warp	Same as warp	Same as warp
2	Warp	Slightly lighter brown than #1, Pantone® 467U	Same as #1	Same as #1
	Weft	Same as warp	Same as #1	Same as #1
3	Warp	Same as #1	Same as #1	Same as #1
	Weft	Same as #1	Same as #1	Same as #1
4	Warp	Light brown, between Pantone® 465U and 466U	Same as #1	Generally, more intense than #1
	Weft	Same as warp	Same as #1	Same as Warp
5	Warp	Same as #1	Same as #1	Same as #4
	Weft	Same as #1	same as #1	Same as #1
6	Fibre only	Off white	Similar to #1, but bluer, Pantone® 247U but more intense	Almost all intense magenta
7	Warp	Light brown, slightly darker than Pantone®465U	Same as #1	Same as #1
	Weft	Same as warp	Same as #1	Same as #1
8	Yarn only	Light brown, between Pantone® 464U and 465U, streaky	Same as #1	Same as #1
9	Fibre only	Same as #4	A little darker than #1, Pantone® 241U	Same as #4
10	Warp	Same as #8	Same as #1	Same as #1
	Weft	Same as #8	Same as #1	Same as #1

Table 22
Phloroglucinol and Hydrochloric Acid Test of Ramie Fibre in Known Specimens

#	Original Colour	Colour Change	Colour Change at 100X
1 Yarn only	White	No colour change	No colour observable
2 Warp	White	No colour change	No colour observable
Weft	White	No colour change	No colour observable
3 Fibre only	White	No colour change	No colour observable
4 Fibre only	White	No colour change	No colour observable
5 Weft only	Off white	No colour change	No colour observable
6 Pile only	Off white	No colour change	No colour observable
7 Yarn only	White	No colour change	No colour observable
8 Fibre only	White	No colour change	No colour observable
9 Yarn only	White	No colour change	No colour observable
10 Yarn only	Off white	No colour change	No colour observable

Zinc Chloroiodide (Herzberg Reagent)

The zinc chloroiodide test causes colour changes in vegetable fibres as well as highlighting the cellular structures within the fibre cells. According to Mathews (1952) zinc chloroiodide causes lignified cellulose to turn yellow and pure cellulose to turn violet. Dislocations or nodes and crossmarkings stain much more intensely. The amorphous nature of these areas allows the stain to penetrate easily.

Strellis and Kennedy (1967) state that while lignified cellulose turns a golden yellow with zinc chloroiodide, extraneous cells and lumina contents stain an intense bright yellow. The reason for this is not given but Batra (1985) does say that when remnants of a protoplast are found in a bast fibre cell, they are usually nitrogenous in nature.

Koch (1963) warns that different methods of reagent preparation cause different colour changes in the same fibre. He says that depending on the method, cellulose can be blue, bluish violet, or reddish violet.

The results of the zinc chloroiodide testing done on known specimens of flax, hemp, jute and ramie fibres are presented in Tables 23 to 26. The Pantone® by Letraset™ colour selector was not used to evaluate colour change because these observations were made under optical microscopy at 100X. The colour changes in the flax fibres vary from deep violet to deep reddish violet (see Table 23). These results are consistent with those reported by the authors in Table 12; however, no pure red colour changes were found. The less intense staining of the #3 weft could possibly be due to poorer penetration of the reagent. This could also account for no staining of the lumina contents. Lignin and extraneous material may have inhibited its passage. However generally, the lumina in flax were made more easily visible by Herzberg reagent.

In spite of hemp testing positive by turning pink for the presence of lignin in phloroglucinol and hydrochloric acid where most flax samples did not, the differences between the colour changes of flax and hemp in zinc chloroiodide are not great (see Table 24). What is important is that staining highlights the numerous crossmarkings in hemp. In specimens #5 and #6 it is possible that less penetration of the staining medium occurred because of lignin and extraneous tissue. These two specimens appear to have been processed only to the stage of retting. Thus, as would be expected, the same intensity of hue is not attained as in the other samples.

The colour changes of jute in zinc chloroiodide show a high degree of consistency. Most fibres turn greenish brown with sometimes some more reddish areas and dark streaked lumina (see Table 25). These results are not consistent with any of the authors' results in Table 12. The staining helps make the nodes, lumina and cell ends visible, characteristics not easily seen without such highlighting. The cell ends visible in jute are particularly unique because, while they taper like those in the other known fibres, their angle is less steep, making the ends considerably more blunt than in the other fibres.

Table 23
Zinc Chloroiodide Test of flax fibres in Known Specimens

#		Original Colour	Colour Change at 100X	Microscopic Observation at 100X
1	Warp	White	Deep violet, some areas are reddish violet, nodes are violet-black Extraneous material - bright yellow	Many nodes in form of X, V, or I, majority without swellings Few lumina visible, show as a fine line
	Weft	White	Same as warp	Same as warp
2	Warp	White	Deep violet, nodes are violet-black Extraneous material - bright yellow	Nodes similar to #1 but with more, larger swollen ones Lumina generally larger and more visible than #1
	Weft	White	Same as warp	Same as warp
3	Warp	White	Same as #2	Same as #2, but with more extraneous material
	Weft	Brown, Pantone® 467U, with lighter and darker flecks	Reddish violet and not stained as intensely as warp Extraneous material - bright yellow	Same as warp, presence of 1 large vessel element-stained blue
4	Warp	Off white	Same as #3	Same as #3
	Weft	Off white	Same as #3	Same as #3 but with parenchyma cells
5	Warp	White	Deep reddish violet, nodes are violet-black Extraneous material - bright yellow	Same as #1
	Weft	White	Same as warp	Same as #1
6	Warp	Off white	Same as #1	Same as #1
	Weft	Off white	Same as #1	Same as #1
7	Warp	White	Same as #5	Same as #1
	Weft	White	Same as #1	Same as #1
8	Yarn only	White	Same as #1, but with more reddish violet	Same as #1
9	Yarn only	Off white	Same as #2	Same as #1
10	Yarn only	Light brown, Pantone® 467U	Same as #2	Same as #1, but with more nodes

Table 24
Zinc Chloroiodide Test of Hemp Fibre in Known Specimens

#	Original Colour	Colour Change at 100X	Microscopic Observation at 100X
1 Yarn only	Beige	Violet-black with some reddish-violet areas and some brown areas, nodes are black Extraneous tissue is violet and bright yellow	Fewer small nodes than generally in flax, but look similar, a few swollen nodes Lumina difficult to see
2 Yarn only	Beige	Same as #1	Same as #1
3 Yarn only	Beige	Same as #1	Same as #1, but can see crossmarkings, they appear as scratch-like marks extending across more than 1 cell
4 Yarn only	Beige	Same as #1, but darker	Same as #3, but crossmarkings not as distinct
5 Fibre only	Dark beige to light brown, Pantone® 466U	Pale violet with some yellow lumina contents, nodes are dark golden yellow	Nodes not as frequent as #1, a few crossmarkings Lumina not visible except where contents stained
6 Fibre only	Same as #5	Violet, pinkish violet, nodes are golden yellow	Nodes not as frequent as #1, a few crossmarkings, has parenchyma cells Lumina difficult to see

The colour changes in ramie were, like jute, quite consistent (see Table 26). They were similar to those of flax, being mostly deep violet or reddish violet as is found in the results of Mathews (1952). Ramie did not have the intense yellow staining of extraneous tissue found in flax. The most unique characteristic observed in the ramie samples treated with zinc chloroiodide was the extreme length of some of the cells. In some cases, a cell with a distinct lumen, highlighted by the stain, could be followed across the whole distance of the microscope slide cover.

Table 25
Zinc Chloroiodide Test of Jute Fibre in Known Specimens

#		Original Colour	Colour change at 100X	Microscopic Observation at 100X
1	Warp	Light brown, Pantone® 466U	Greenish brown with some areas more red, some more yellow, dark streaky lumina contents	Nodes appear mostly as bends Lumina often appear broken open and vary in thickness, many contain air
	Weft	Same as warp	Same as warp	Same as warp
2	Warp	Slightly lighter brown than #1, Pantone® 467U	Same as #1 but without red	Fibres more transparent than #1, nodes visible often across all cells in a fibre, has tapered cell ends within bundles Lumina same as #1
	Weft	Same as #1	Same as warp	Same as warp
3	Warp	Same as #1	Same as #1	Has all characteristics of #1 and #2, but can see no cell ends
	Weft	Same as #1	Same as #1	Same as warp
4	Warp	Light brown, between Pantone®465U and466U	Same as #1	Same as #1 and #2
	Weft	Same as warp	Same as #1	Same as #1 and #2
5	Warp	Same as #1	Same as #1	Same as #1 and #2
	Weft	Same as #1	Same as #1	Same as #1 and #2
6	Fibre only	Off white	Very dark greenish-brown with, dark streaky lumina contents	Same as #1 and #2
7	Warp	Light brown, Slightly darker than Pantone® 465U	Same as #1 with a lot of reddish brown	Same as #1 and #2
	Weft	Same as warp	Same as #1, but more yellow	Same as #1 and #2
8	Fibre only	Light brown, between Pantone® 464U and 465U, streaky	Same as #1	Same as #1 and #2
9	Fibre only	Same as #4	Same as #1, but more yellow	Same as #1 and #2
10	Warp	Same as #8	Same as #1	Same as #1 and #2
	Weft	Same as #8	Same as #1	Same as #1 and #2 with some very distinctive nodes

Table 26
Zinc Chloroiodide Test of Ramie Fibre in Known Specimens

#		Original Colour	Colour Change at 100X	Microscopic observation at 100X
1	Yarn only	White	Very deep red-violet, nodes are violet-black	Many nodes in various shapes Lumina difficult to see
2	Warp	White	Same as #1 with some even more deeply stained	Same as #1 Some wide lumina visible, some are straight, some uneven
	Weft	White	Same as warp, but a little less red	Same as warp
3	Fibre only	White	Deep violet, nodes violet-black	Many nodes, many are large and swollen or bent (elbow like) Some wide lumina visible, very straight
4	Fibre only	White	Same as #3	Same as #3, a few less large swollen nodes Has some narrow lumina as well as wide
5	Weft only	Off white	Same as #1	Same as #4
6	Pile only	Off white	Brownish purple, nodes black	Same as #3, but also has some narrow lumina
7	Yarn only	White	Same as #6	Same as #6
8	Fibre only	White	Same as #1	Same as #4
9	Yarn only	White	Same as #6, very slightly paler	Same as #6
10	Yarn only	Off white	Same as #1, but more violet	Same as #4

Cuprammonium Hydroxide (Schweitzer's Reagent)

Cuprammonium hydroxide is a swelling and dissolving agent for cellulose. Flax, hemp, jute and ramie each have characteristic swelling and dissolution patterns. Preston

(1942) notes that the observation of swollen fibres can be misleading for the study of cell wall structure in bast fibre cells; for instance, angle, but not direction of fibrillar orientation can be changed by the mechanism of swelling and by any pressure inadvertently put on the slide cover slip. The number of cell wall fissures can also change under swelling. It is important to concentrate on typical reactions in each fibre type rather than on specific accentuated or deformed cell wall structures.

Although the precise mechanism for cuprammonium hydroxide can not be given, Feller, Lee and Conran (1985) state that the solvent power of alkali metal hydroxide solutions at moderate temperatures is related to their ability to swell cellulose fibres. Maximum solubility can be obtained at about the same concentration as maximum swelling.

The method used for swelling the specimens with cuprammonium hydroxide has been described in Chapter 3. Observations were made at 100X and 400X switching back and forth continually between the two powers as swelling and dissolution progressed.

Flax.

The swelling and dissolution of the flax fibres in cuprammonium hydroxide was extremely rapid, taking usually only 30 seconds to one minute. Initially, the fibre cells were swollen laterally and shrunk longitudinally. Often before fibrillar orientation became visible, strong lines, parallel to the fibre axis could be seen in the cell walls. Hock (1942) states that these lines that he calls lamellae consist of alternating layers of strongly and weakly birefringent material. At the same time the lumen became visible. As the wall continued to swell, the nodes protruded outward as the lumen became wider. Then the cell started to fold, accordion-like, at the nodes. It was possible, at this stage, to see the fibrillar orientation of the cell as it broke down (see Figure 6). In all cases, the orientation was in an S direction. As shown in Figure 5a, Hock (1942) states that this is the secondary cell wall; the primary cell wall is always oriented in a Z direction, though it rarely can be seen. He says that it appears all subsequent layers are oriented in an S direction, though Anderson (1927) does not concur, saying that layers alternate their directions.

The next stage was complete dissolution of the cell with only a snake-like thread of protoplasm left from the lumen. This thread was dramatically visible and unique to flax in the cuprammonium observations. Sometimes the protoplasm appeared as a small amount of granular material rather than a thread. In either case, after 24 hours most protoplasmic material was also dissolved. Regardless of the time required for dissolution of the cell and its contents, a transparent, pale blue viscous residue remained.

Vary rarely, ballooning occurred. It is possible, as described by Maiti (1980), that this was caused by areas of the primary cell wall that remained resistant to swelling in the solvent longer than did the secondary cell wall. Hock (1942), however, states that the primary cell wall is attacked along with the intercellular material during retting.

In the less processed fibres, the swelling and dissolution reactions of the fibre cells were essentially the same with more extraneous debris remaining after the dissolution had taken place. In some areas the reaction took several minutes as penetration of the solvent was not as easily facilitated.

Hemp.

The swelling and dissolution of hemp fibres in cuprammonium hydroxide occurred at a much slower rate than that of flax, as predicted by Heyn (1954). As the hemp was almost entirely in bundles, the first occurrence after the addition of the reagent was the

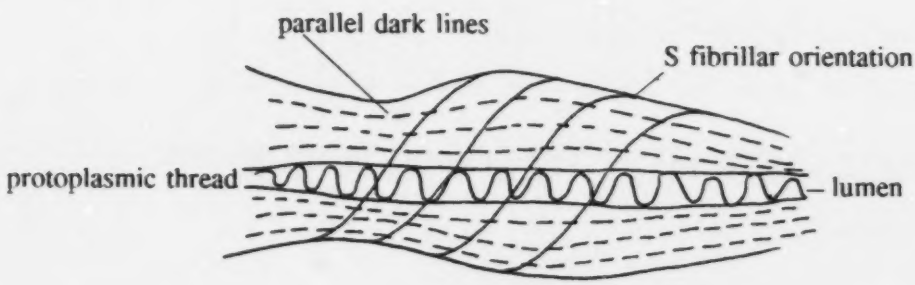


Figure 6. The swelling of a flax cell (area between nodes) in cuprammonium hydroxide.

peeling away of individual cells from the bundle. They would then swell slowly and usually dissolve, taking several minutes. It is difficult to put a time frame around the process because if the fibres were not completely dissolved after several minutes, reactions stopped. Upon addition of more reagent, the reaction once again continued. Also, while the reagent reacted with the outside fibres in a bundle, it also slowly entered the ends of the bundles. After 24 hours, though the hemp fibre cells were dissolved, barely visible ruffled lines were still present. As with flax, a transparent pale blue viscous residue remained.

In all cases, with hemp, there was some ballooning such as that shown in Figure 7. Either middle lamellar material or the primary cell wall which was less flexible and slower to dissolve than the cellulose in the cell wall, caused spiral constrictions. The fibrillar orientation of the hemp fibre cells was not as easy to see as that of flax. When visible, it was always in a Z direction which is consistent with figure 5b.

There were some dramatic differences between the reactions of hemp and flax. As Heyn (1954) has predicted, no snakelike thread of protoplasm was in the lumina of hemp. In fact, there was no visible protoplasm at all, though a lumen was sometimes visible. Constriction of the lumen can occur when the cellulose wall is forced to expand inward because of the constricting external layer present around most hemp fibre cells (Hearle & Peters, 1963). The lumina once again opened up as the cellulose dissolved. The resistant middle lamella which contained lignin became ruffled and pleated. As the fibre cell shrank longitudinally, the middle lamella did not. This concurs with the observations of Kundu and Preston (1940) and Heyn (1954). This characteristic was dramatic and unique to hemp in the cuprammonium observations.

Jute.

The swelling and dissolution of jute fibres in cuprammonium hydroxide occurred at an even slower rate than that of hemp, taking usually several minutes longer. The initial reaction took place in the most external cells in a bundle causing them to swell and pull away from the bundle as can be seen in Figure 8. Hearle and Peters (1963) state that in jute, lignin is present in higher proportions in the inner layers of the cell wall after retting

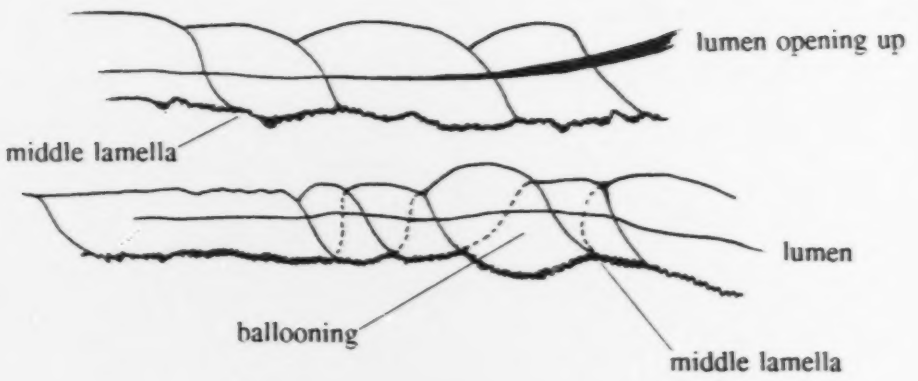


Figure 7. The swelling of hemp in cup[illegible]mmonium hydroxide.

than in hemp, causing swelling to be manifested by an increase in the external dimensions of the cell. Jute is generally more lignified than flax, hemp or ramie (Florian, 1990).

As the fibre cells pulled away from the bundle, they took a strip of the middle lamellar material with them. The cell twisted in the direction of the fibrillar orientation causing the unswollen middle lamella to wrap around the cell as it twisted. Preston (1942) states that the constricting spiral band is the resistant primary cell wall. However, when cells remain in bundles after retting, it seems reasonable to assume that the lignified middle lamella will still be in place between cells in the bundle. As the spiral constricting band was quite dense, it is possible that it was lignified middle lamella. The unique spiral ballooning that occurred was not unlike the appearance of a unicorn horn with the pointed tip being the cell end. The angle of orientation was in a Z direction. This was easy to see and became apparent almost immediately.

Sometimes, several fibre cells that had broken away from the bundle would wrap around each other creating the appearance of a many-strand braid. This formation was also unique to jute in this testing.

Unlike hemp and flax, after 24 hours, some remnants of the jute fibre cells remained on the glass slide. Besides being visible with a microscope, they could be seen with the naked eye as could the pale blue transparent cellulose residue.

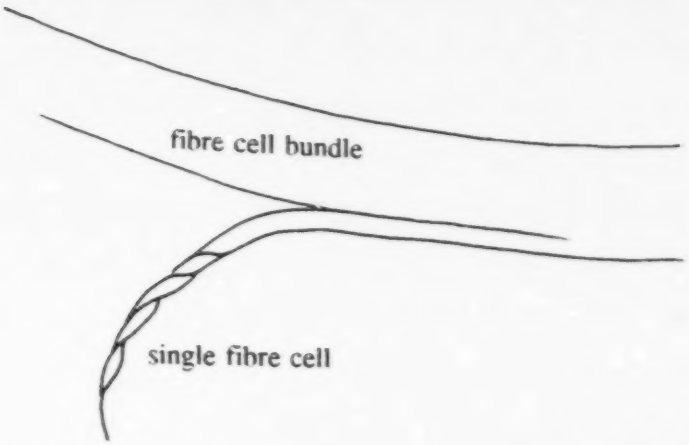


Figure 8. The swelling of jute in cuprammonium hydroxide

Lumina were not easily visible in the jute fibre cells except when they contained air. The lumina containing air bubbles were all broad and even sided. A few lumina could be seen containing protoplasm remains. These lumina were narrow and uneven.

Ramie.

No detailed descriptions of the behaviour of ramie fibre when swollen and dissolved with cuprammonium hydroxide could be found. One reason for this could be that it is a more straightforward process than with the other fibre species in this study. In any case, dissolution occurs so quickly that the fibre cells could not be observed swelling. Most samples were completely dissolved in thirty seconds, while a few which contained larger, more mature cells took about one minute and had swollen slightly first. The rapidity of dissolution was probably due to the high degree of purity of cellulose in ramie. All samples left a transparent viscous blue residue.

The S direction of fibrillar orientation was at a more acute angle to the fibre axis than were the fibrillar orientations of flax, hemp and jute. This was probably due to the small amount of swelling taking place. Within the 30 second period required to dissolve most fibres, they followed the same pattern, which was unique to ramie in this study. First, each fibre cell became uneven as the solvent entered and dissolved the more amorphous nodes. The fibre became thinner in the node area and then broke off into

islands of cell between where the nodes had been. The lumen in these islands then opened up and dissolution appeared to progress from both the inside and the outside of the fibre cell.

To summarize, it can be seen from the above observations on the swelling and dissolution of flax, hemp, jute and ramie that they all followed patterns that had similarities as well as great differences. It is the uniquely different reactions of the four species to the cuprammonium hydroxide that are the most useful in determining the identity of unknown specimens. The thread-like remnants of protoplasm left after dissolution of flax fibres are a unique characteristic. In hemp, the ruffling of the middle lamella sets it apart from the other three fibres when it swells and dissolves. Jute has unique twisting individual cells that break partially away from bundles. Ramie dissolves more rapidly than the other fibres, so rapidly that swelling rarely has time to occur.

The Twist Test

There is some confusion in the literature regarding the direction of twist observed for flax, hemp, jute and ramie in the twist test when single fibres are wet out, then allowed to rotate freely upon drying. Hock (1942) is clear that when observing the fibres twist they should be viewed from above. When this is done, flax and ramie turn counterclockwise while hemp and jute turn clockwise. Schaffer (1981b) states that when the viewer looks down the fibre from the free end, flax and ramie turn clockwise and hemp and jute turn counterclockwise. Kirby (1963) and Strellis and Kennedy (1967) concur with this but do not give the directions for all four fibres. Neither The Textile Institute (1975) nor Goodway (1987) give directions for how to view the fibres. Both report that flax and ramie turn clockwise and hemp and jute turn counterclockwise.

The twist test according to Hock (1942) was administered to the known specimens as described in Chapter 3. The results of these tests were 100 per cent consistent and coincided with those of Hock.

Flax fibres, in all cases, rotated counterclockwise. The speed of revolution varied somewhat. It is difficult to draw conclusions about this variation because there were slight inconsistencies in length and thickness of the fibre samples and in their degrees of

processing. It was sometimes necessary to submerge the fibres in water more than once to get a reaction. No reason for this could be determined and no meaningful pattern in this behaviour could be discerned. For instance, it was not necessarily the more lignified fibres that took two submersions to wet out. Also, specimens #5, #9 and #10 were all slower to rotate than the other fibre. While specimens #9 and #10 were slightly less processed than the other specimens (except for the weft of specimen #3), specimen #5 was highly processed. The weft of #3, though slightly lignified and less processed than most of the specimens turned as rapidly as the highly processed specimens. Neither degree of processing nor degree of lignification appear to determine consistently the amount or speed of twist.

The hemp fibres all rotated clockwise. Specimen #5 revolved very slowly and slightly compared to the others. This specimen was the most lignified of the hemp. As in flax, unavoidable inconsistencies in sample length, thickness and degree of processing could have been a contributing factor. It was necessary, in some cases, to submerge the fibres more than once to obtain a response.

The jute fibres all rotated clockwise. Unlike flax and hemp, they consistently moved quite rapidly. There was little variation in speed. In many cases, twisting was so dramatic that it was possible to see the fibres turning counterclockwise in the water as they were being wet out. Inconsistencies in sample length, thickness and degree of processing appeared to have no effect on speed of rotation.

The ramie fibres all rotated counterclockwise with the exception of specimen #6 which was not tested. The only portion of this specimen that was ramie was the pile which was too short for this test. The other specimens, like jute, showed little variation in speed. They all turned quite rapidly, almost as quickly as jute.

Fibre Diameter Measurements

Fibre diameter measurements were taken of the flax, hemp, jute and ramie specimens as well as of the unknown specimens in the manner described in Chapter 3. The frequencies are shown in histogram form in Appendix A-2. Both the knowns and

the unknowns histograms are kept together in Appendix A-2 for easy comparison with each other.

Frequencies of fibre diameters.

The largest number of flax fibres were found to be between 15.0 μm and 20.0 μm thick with many of the fibres having a diameter of between 5.0 μm and 20.0 μm. The flax fibres were unicellular, having a total range of from under 5.0 μm to 40.0 μm. A total of 850 fibres were measured.

Hemp fibres, unlike those of flax were found in bundles and had a much broader range of thickness measuring from between 0.0 μm and 50.0 μm up to 400.0 μm. By far, the largest number of diameters occurred under 50.0 μm. This created an overlap with the flax fibres, as all the flax fibres were under 40.0 μm in diameter. Therefore, flax and hemp showed some similarity in diameters, although the flax fibres were unicellular and the hemp fibres occurred in bundles. A total of 350 fibres were measured.

Jute fibres, like hemp, were found in bundles. The largest number of jute fibres was between 25.0 μm and 50.0 μm thick. The overall range of jute fibre diameters was from between 0.0 μm and 25.0 μm up to 200.0 μm. This range was within the range of hemp and overlaps with that of flax. The jute fibre diameters are closer in thickness to those of hemp than to those of flax. A total of 850 jute fibres were measured.

Ramie fibres, like flax were found as single fibre cells. The largest number of ramie fibres measured between 10.1 μm and 20.0 μm. The overall range was from under 10.0 μm to 80.0 μm, twice as extensive as that for flax. Ramie diameters overlap with those of flax, but the majority of their diameters range up to 40.0 μm rather than to 20.0 μm as for flax. A total of 550 fibres were measured.

The fibre diameter measurements done in this study can be compared with those in the literature. Flax, measuring up to 40.0 μm, compares almost exactly with the measurements of Florian (1990). Catling and Grayson (1982) and Peters (1963) have ranges within the flax fibre diameter range in this study.

Heyn (1954) states that hemp has a greater variety of diameter measurements than flax which is born out in this study even though Heyn was measuring single cells. The standard deviation for hemp indicates an extremely large variation in diameter measurements; much greater than the other three fibre specimens. Florian (1990) and Catling and Grayson (1982), all measuring single fibre cells, show figures well below 100.0 μm for hemp diameters which are within the range of the majority of those found in this study.

Jute fibre diameters were all under 100.0 μm. Catling and Grayson (1982), Florian (1990) and Heyn (1954) had measurements within this range, but the widest measurement was 32.6 μm, much narrower than many of those done in this study. These authors were measuring single fibre cells.

Heyn (1954) states that flax has a much smaller diameter than does ramie. The measurements of Florian (1990) and Catling and Grayson (1982) concur with this as do those done in this study. The range of the majority of the fibre diameters is lower than those ranges found in the literature.

Means and standard deviations of the fibre diameters.

Table 27 gives the means and standard deviations calculated from the fibre diameters of the four known species.

Table 27
Fibre Cell Diameter Means and Standard Deviations (μm)

Fibre	Mean ($\bar{X}$)	Standard Deviation (σ)
Flax	14.7	5.3
Hemp	58.7	69.7
Jute	54.7	27.7
Ramie	21.6	9.5

Flax and ramie, being single cell fibres have the smallest diameter means of the four specimens at 14.7 μm for flax and 21.6μm for ramie. Ramie is thicker than flax and has a greater standard deviation. When observing ramie under optical microscopy, it can be seen that there is a greater variety of fibre sizes as well as occasional very large fibre cells with knurled nodes like those seen in c, Plate 4. Catling and Grayson (1982) give means somewhat greater than those found for this study.

Hemp and jute both have means that are very close in size to each other at 58.7 μm for hemp and 54.7 μm for jute. Hemp has a standard deviation which is much greater than any of the other fibres, in fact, it is greater than the mean for hemp. This, once again, demonstrates the vast variation possible in the diameter measurements of hemp fibre. Means from Catling and Grayson (1982) and Florian (1990) are lower than in this study, but they measured single cells rather than cell bundles.

SEM Analysis

The photographs taken of flax, hemp, jute and ramie in plates 1, 2, 3, and 4 are typical of each type of fibre. The first two photographs on each plate show the general structures of each fibre type, with the second one being at a higher magnification than the first. The third photograph shows structural detail.

It can be seen in photograph (a) of Plate 1 that there is some variety in fibre diameter in flax. Even so, it should be mentioned again that flax had the smallest standard deviation of the four species in this study at 5.3 μm. All three photographs in this plate show the unicellular quality of most of the flax specimens as well as their typical bamboo-like appearance caused by frequent swollen nodes. In (c), a strip of either primary or secondary cell wall is lying along a fibre cell. This is a common occurrence and probably the result of mechanical action.

The photographs of hemp in Plate 2 show two striking differences from flax; the fibre cells are almost all in bundles, some of which are very large, and the character of the nodes is quite different. There are fewer of them and they appear together across several fibre cells. When nodes are seen at bends, the suggestion is that they are caused mechanically, possibly in processing. It should be mentioned that SEM creates essentially

a topographical record. Interior structures of the cell can not be seen. Therefore, only nodes of the swollen type are visible here as they are in flax, jute and ramie. In (c), three nodes on three fibre cells are visible. The irregular appearance of the cell wall surface is probably caused by attached middle lamellar material.

Plate 3 shows jute with the fibre cells in bundles. Where there appear to be single cells, these cells are, in fact, anchored into bundles at one end. Nodes are barely visible as jute has fewer nodes than the other three fibre species and almost all are of the unswollen type. Two nodes can be seen in the centre of (c), immediately below the remnants of a vessel element. It is difficult to see the unique characteristics of jute using SEM, such as cell ends and wide lumina with air bubbles.

The photographs of ramie in Plate 4 show both similarities to and great differences from flax. Ramie does occur in single cells with slightly greater size variety, as can be determined by its standard deviation of 9.5 μm, and has visible nodes. However, the overall appearance is not bamboo-like when viewed under SEM. The nodes sometimes are very swollen and can take on grotesque shapes as shown in Plate 4, b and c. A common and unique occurrence in ramie is flat, twisting, ribbon-like cells. These are caused by the collapse of a particularly wide lumen as can be seen in the bottom of Plate 4 a.

Plates 5 and 6 give a selection of six of the unknown specimens. Photograph (a) in Plate 5 shows a fibre bundle with large amounts of extraneous material. Impressions of parenchyma cells can be seen on the surface of the fibre with some attached cell walls still present. The fibres in Plate 5 b show single fibre cells with the typical bamboo-like appearance of flax. The S fibrillar orientation is indicated by the fissure present in the cell second from the top of the photograph. Presence of fine debris partially obscures the fibre in Plate 5 c. The spheres in the right centre top of the photograph are probably insect eggs. These fibres show swollen nodes of the type found in ramie. However, they consist of bundles, not single cells like those of ramie.

Plate 6 shows three examples of degraded fibres. It is possible that the areas of degradation in the large fibre cell in Plate 6 a are due to microbial entry through the pits in the cells. The areas around the pits would be degraded first. A degraded node can

be seen in this cell. The smaller cell to the right shows degradation to the extent that the cell wall has collapsed and is open to the lumen. Extreme encrustation and degradation is evident in Plate 6 b. True size of bundles or fibre cells can not be seen. The break in the encrusted mass on the left indicates brittleness and breaking caused by extreme shortening of the cellulose polymer. Several fungal hyphae can be seen in Plate 6 c. The one on the right does not twist, but lies flat along the fibre cell. It crosses a node near the bottom. Another can be seen at the left centre of the photograph. This photograph also shows a large amount of debris.

PLATE 1

a. Flax specimen #2. Fibre cells showing variety in size. Scale bar = 40 μm

b. Flax specimen #1. Fibre cells with swollen nodes. Scale bar = 40 μm

c. Flax specimen #5. Fibre cell showing nodes and strip of primary or secondary cell wall. Scale bar = 20 μm

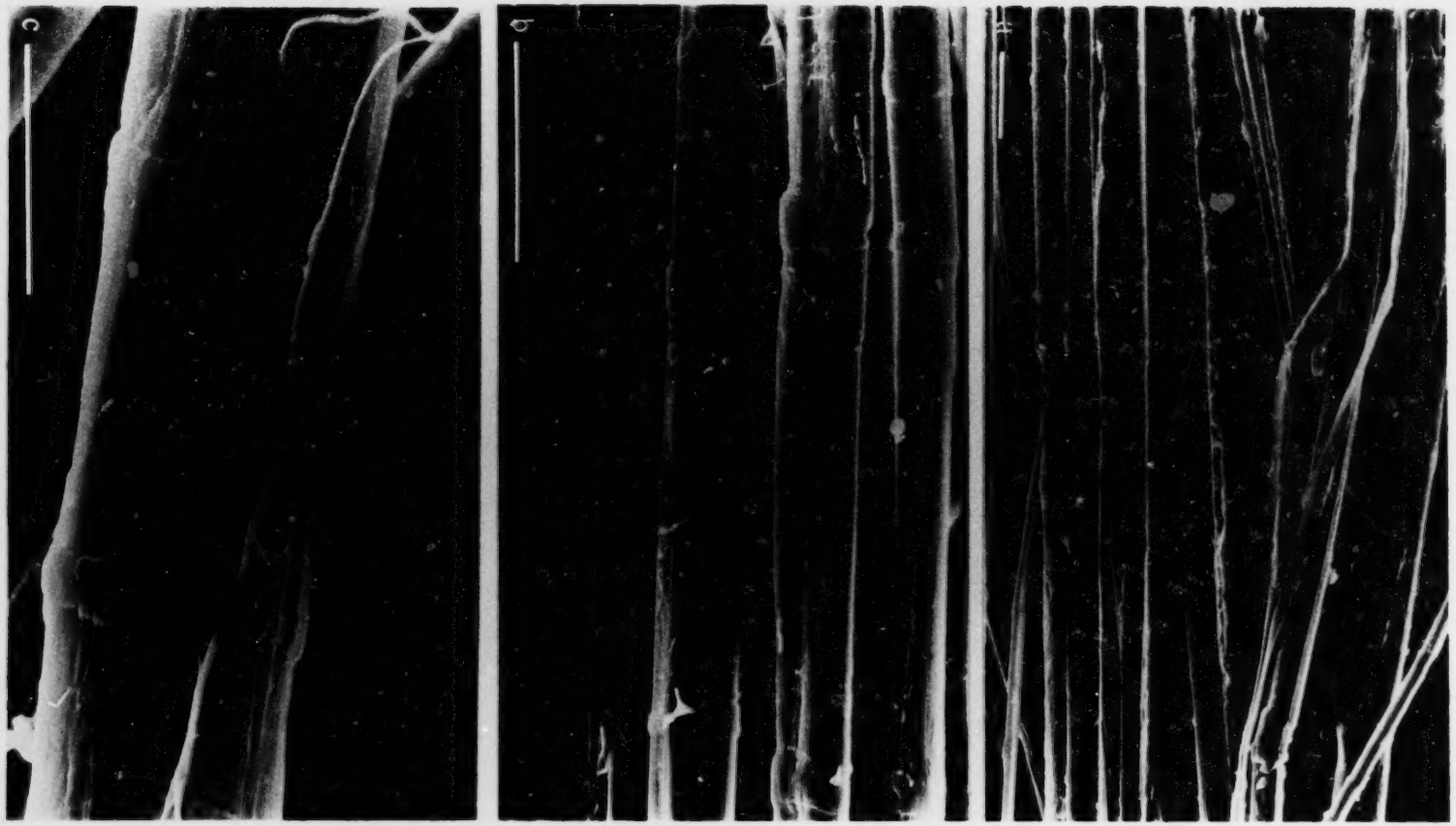


PLATE 2

a. Hemp specimen #5. Fibre cell bundles with bundle showing nodes across entire width. Scale bar = 40 μm

b. Hemp specimen #2. Fibre cell bundle showing cells with nodes at bend and extraneous tissue. Scale bar = 40 μm

c. Hemp specimen #6. Fibre cells with nodes showing part of a bundle. Scale bar = 20 μm

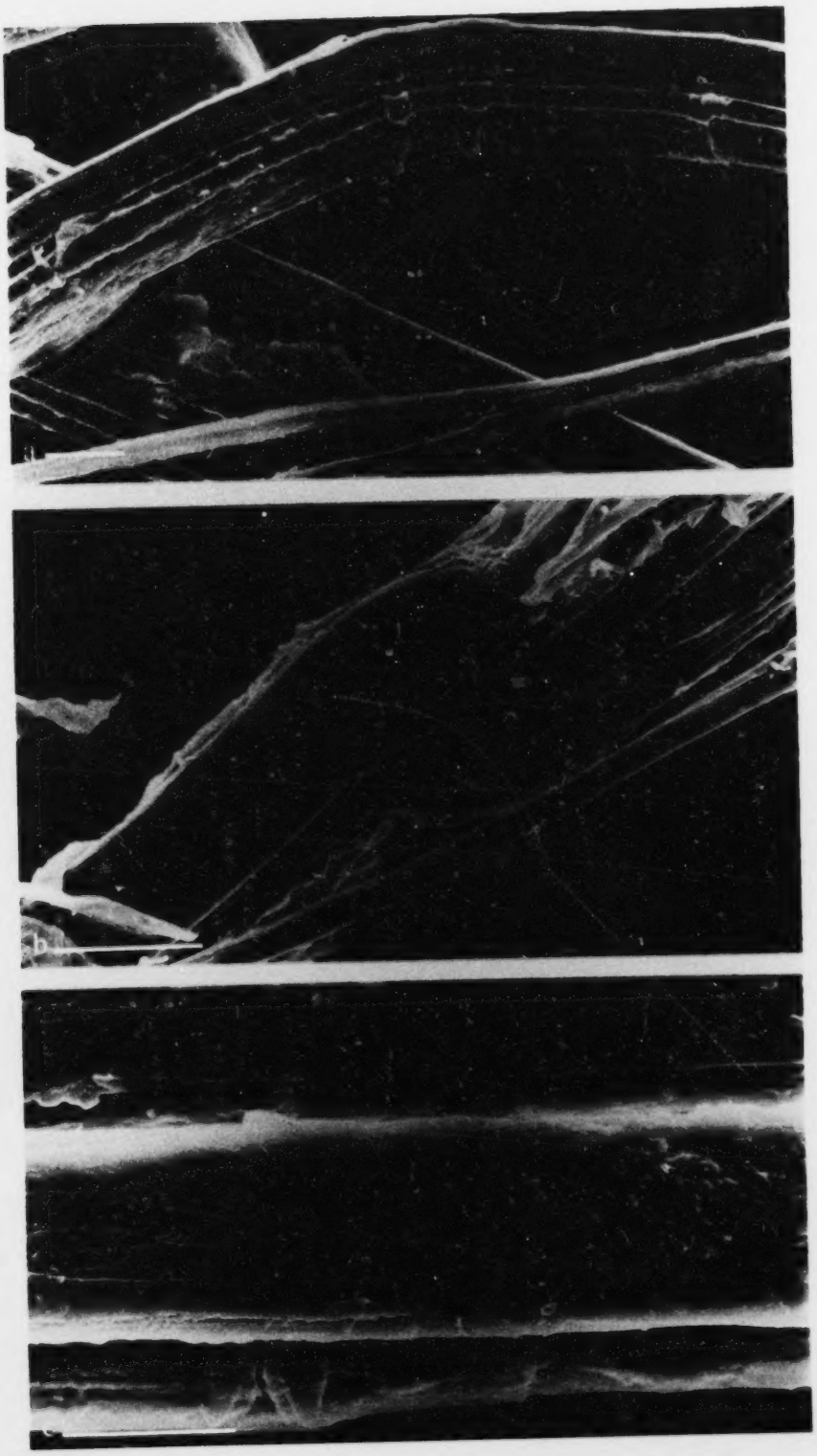

b
c

PLATE 3

a. Jute specimen #4. Fibre cell bundles with extraneous tissue. Scale bar = 40 μm

b. Jute specimen #1. Fibre cell bundles. Scale bar = 40 μm

c. Jute specimen #10. Fibre cell bundle with portion of xylem element in centre of photograph. Scale bar = 50 μm

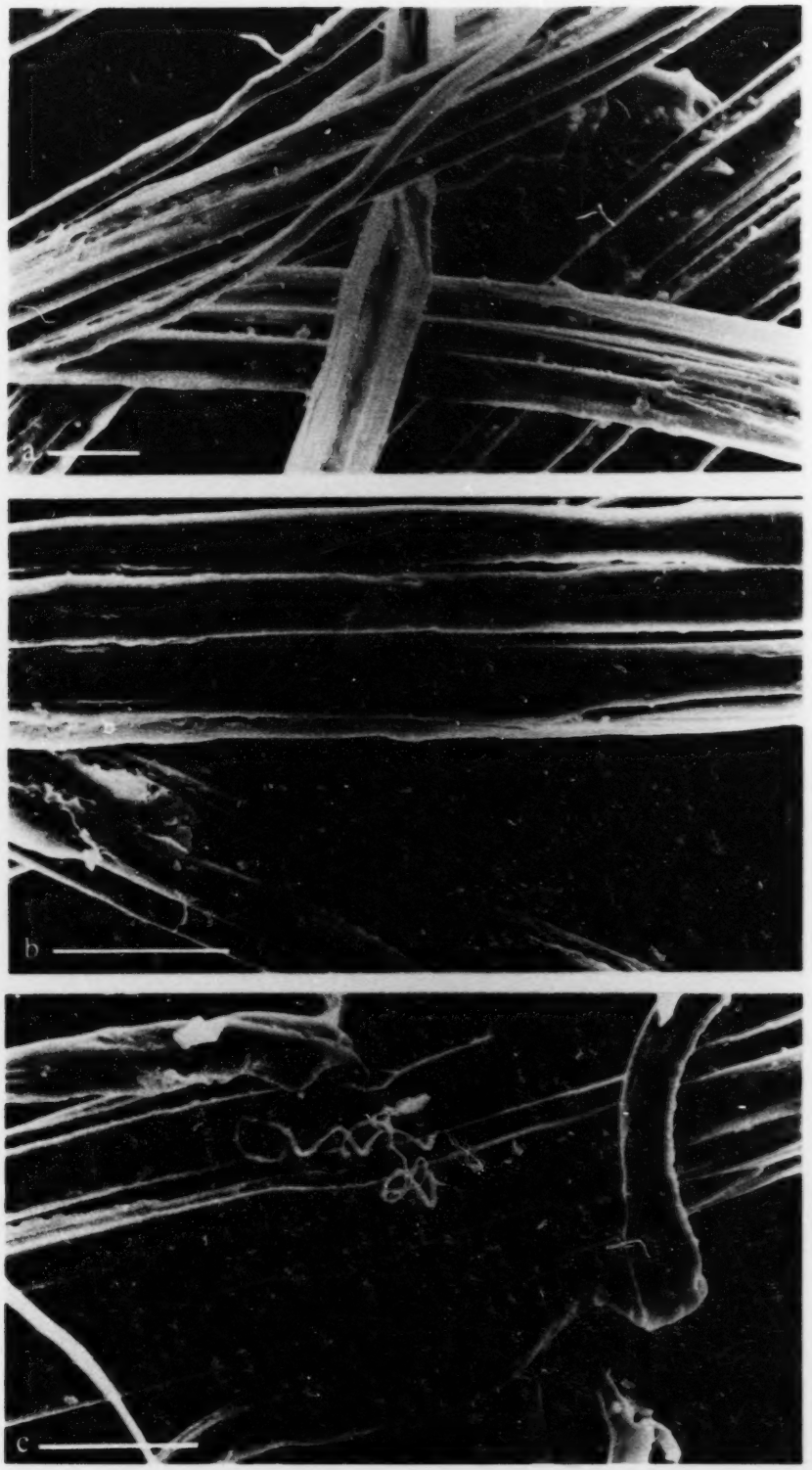

a
b
c

PLATE 4

a. Ramie specimen #1. Fibre cells with swollen nodes. Cell at bottom of photograph is collapsed and twisted. Scale bar = 40 μm

b. Ramie specimen #4. Fibre cells with nodes. Scale bar = 20 μm

c. Ramie specimen #4. Fibre cell node. Scale bar = 20 μm

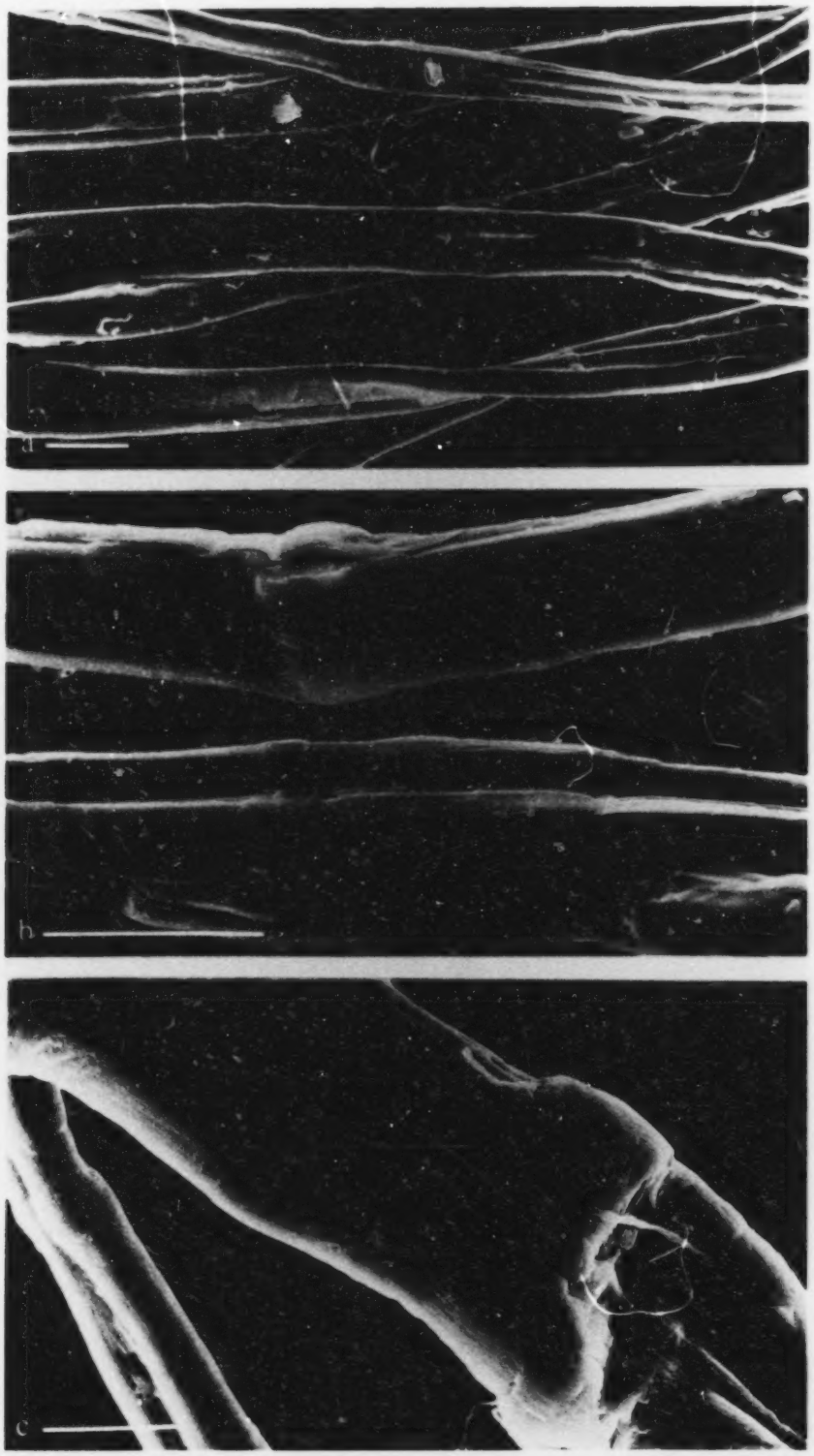

a
b
c

PLATE 5

a. Unknown specimen #10. Fibres from Royal Ontario Museum. Remnants of adjacent parenchyma cells are attached to fibre cell bundle. Scale bar = 40 μm

b. Unknown specimen #7. Fibres from Egyptian Fabric, Royal Ontario Museum. 1450 B.C. Fibre cells with nodes. Fibrillar orientation in S direction is visible in cell second from top. Scale bar = 40 μm

c. Unknown specimen #12. Fibres from Royal Ontario Museum. Small fibre cell bundles with visible nodes. Fibres are covered with fine debris. Spherical shapes in top right half of photograph are possibly insect eggs. Scale bar = 40 μm

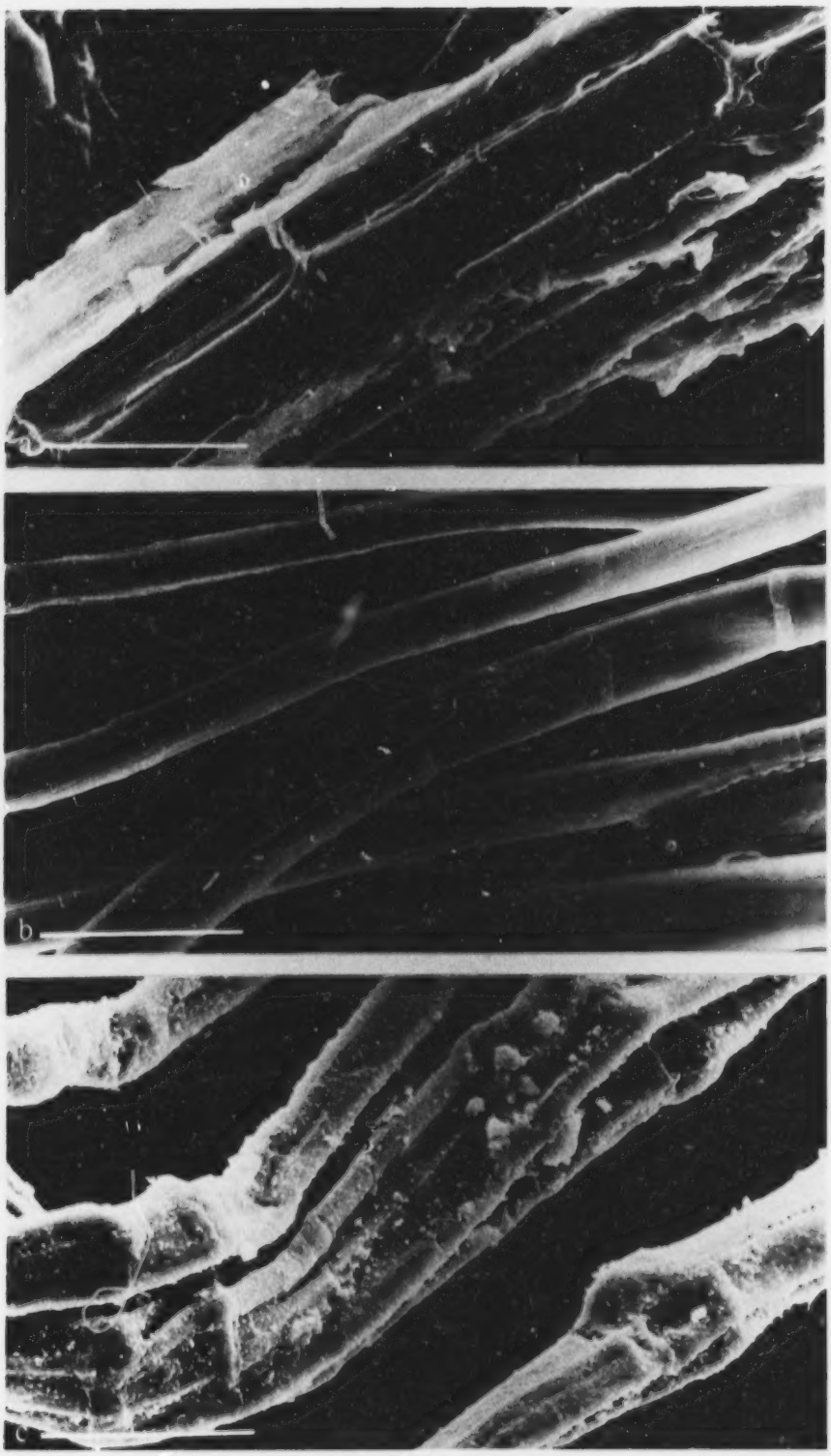
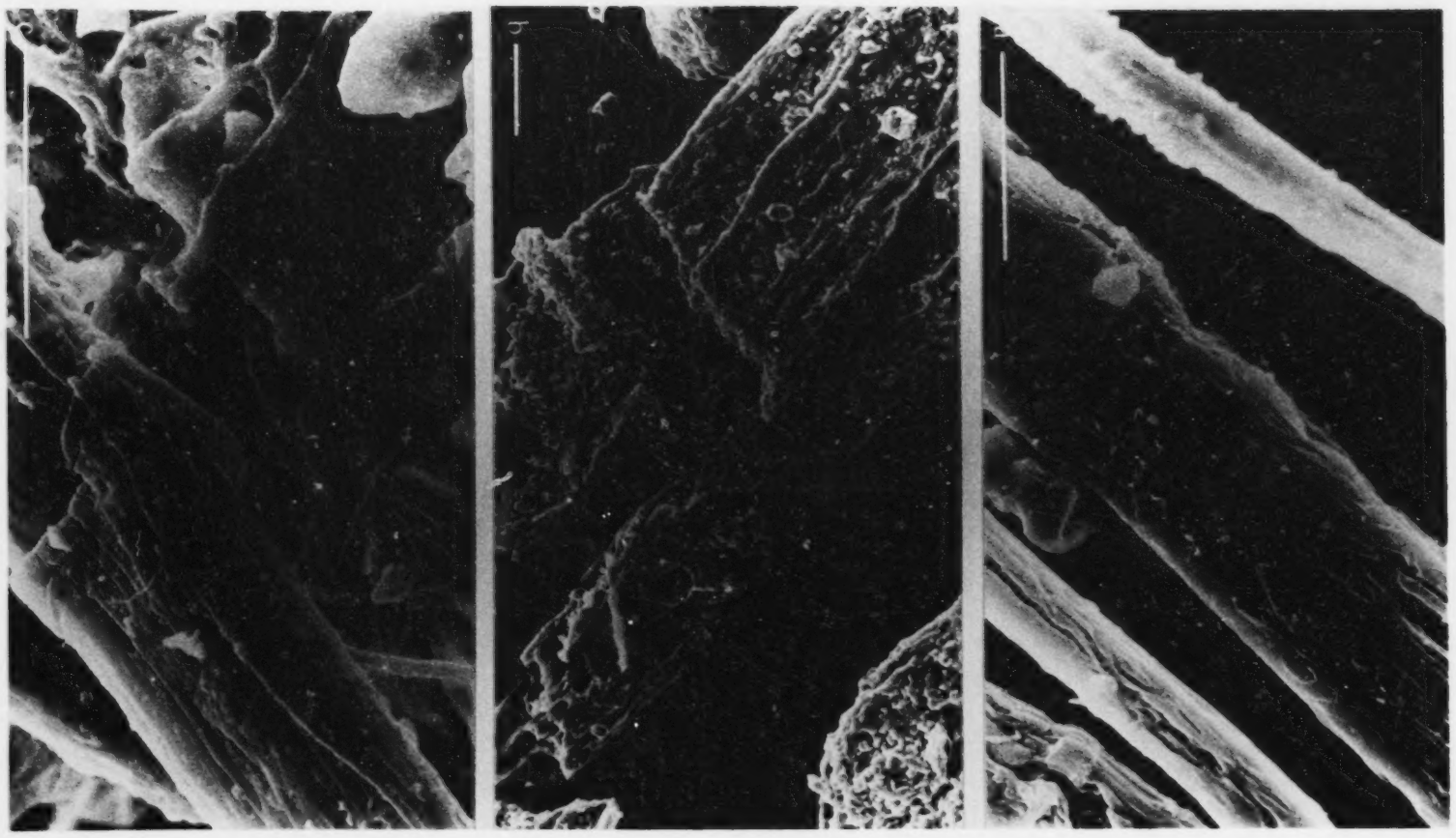

a
b

PLATE 6

a. Unknown specimen #14. Canvas from Franklin Collection. Fibre cells are very degraded with cell wall of cell on right collapsed into lumen. Cell in centre has one node and some debris. Scale bar = 20 μm

b. Unknown specimen #16. Cordage from Franklin Collection. Fibre cells are extremely degraded with encrustations and large amount of debris present. Break across axis of fibre cell "bundle" indicates brittleness and extreme shortening of cellulose polymer chain. Scale bar = 50 μm

c. Unknown specimen #19. Canvas from Franklin Collection. Fibre cells are degraded with encrustations and large amount of debris present. Fungal hyphae are visible throughout with one crossing a fibre cell node near bottom centre of photograph. Scale bar = 20 μm



EDXA

Energy Dispersive X-Ray Analysis (EDXA) was performed on crystals found on the surface of four of the specimens. Two hemp samples with crystals were analyzed as well as crystals from two of the unknown specimens. The crystals were salts high in calcium content. They may be calcium oxalate crystals because Jakes and Angel (1989) state that calcium oxalate crystals form in plants as they grow and that large amounts of calcium indicate bast fibres. They go on to say that long after other parts of a fibre have been destroyed, these crystals will still be present. Rahman (1979), however, states that crystals containing calcium are lignin based and that when lignin is removed, so too, are the crystals. Jakes and Angel (1989) do say that calcium can be found on fibres after soap cleaning or retting in hard water. In any case, calcium in crystals was often found on the surface of the bast fibres in this study. Whether they are a common occurrence on other plant variety fibres was not established.

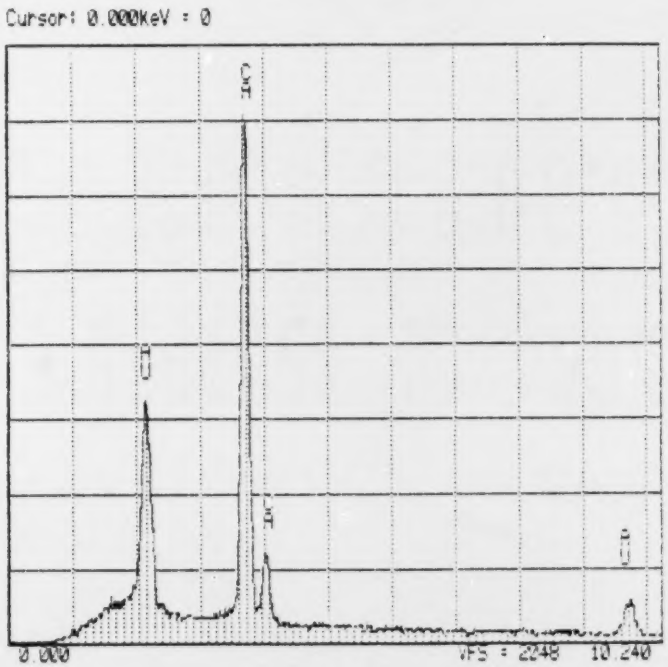


Figure 9. EDXA graph showing high level of calcium present in crystal from hemp specimen # 3.

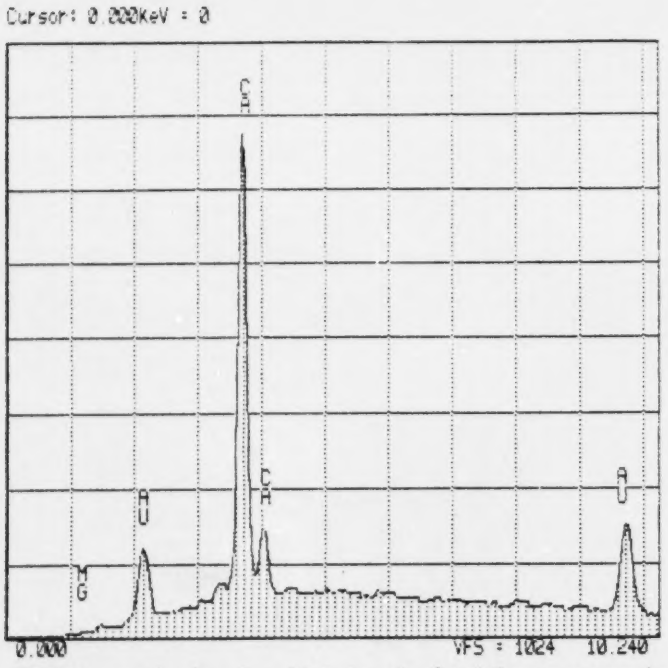


Figure 10. EDXA graph showing high level of calcium present in crystal from hemp specimen #6.

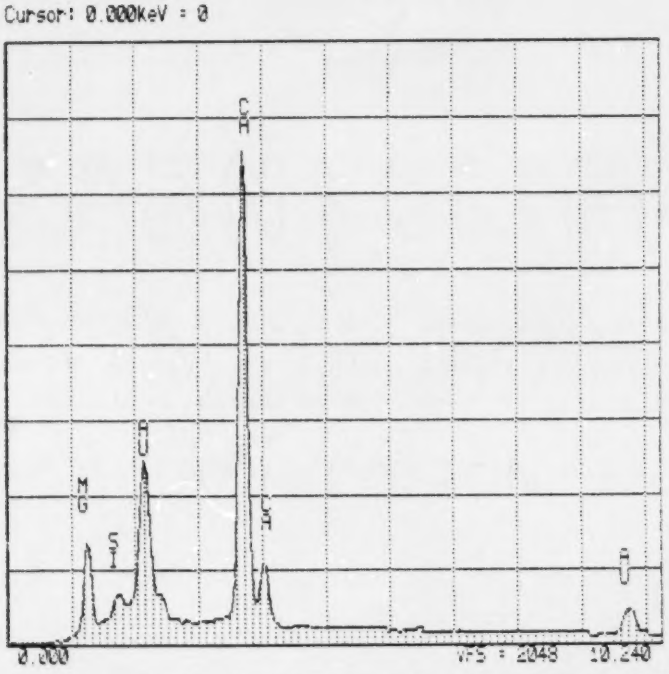


Figure 11. EDXA graph showing high level of calcium present in crystal from unknown specimen #4, canvas from CCI.

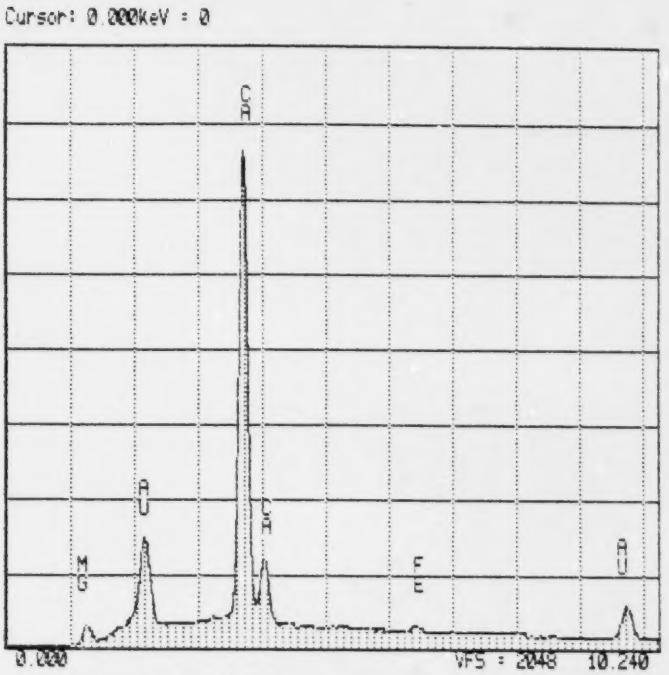


Figure 12. EDXA graph showing high level of calcium present in crystals from unknown specimen #15, canvas from the Franklin Collection.

Testing of the Unknown Specimens

The twenty unknown specimens were tested with phloroglucinol and hydrochloric acid, Herzberg reagent and Schweitzer's reagent. Most specimens were administered the twist test, however, some were too degraded and encrusted to be included. For the same reason, several of the specimen diameters could not be measured. Microscopic observations were done under the same magnifications as were the known specimens. All were observed under SEM.

For general observations and chemical testing, the warp and weft of the fabric specimens were tested as one because the specimens were very small. However, in the fibre diameter measurements, an assigned warp and weft were measured separately as two specimens where possible. This was done because it was the last observation made in the study and enough of each specimen remained to carry out the measurements in this way.

Results of the Unknown Specimens

The following observations were made of the unknown specimens. These are reported in table form, with one table for each specimen. The interpretation and possible identification of each fibre is also given and takes into consideration the initial microscopic observations made in Table 18 and the diameter frequencies in Appendix A-2.

Because the observations of the known specimens were consistent, the assumption has been made that they can be used as standards for comparing the results of the unknown specimens. The possibility that the unknown specimens are neither flax, hemp, jute nor ramie must be borne in mind, however, for them to be truly unknowns.

Specimen #1 contained no lignin as no colour change took place when it was tested with phloroglucinol and hydrochloric acid. The reddish brown colour that resulted from the Herzberg reagent is not clearly indicative of any of the four knowns, but the yellow extraneous material is most typical of flax and hemp. The S fibrillar orientation and counterclockwise twist are consistent with flax and ramie. The pattern of swelling and dissolution as well as the fibre diameter means, standard deviations and frequency distributions are closest to those of flax. These attributes along with the overall bamboo-like appearance of the nodes and the fact that the textile is a white napkin support the identification of flax.

Because there were many gradations of colour change, varying from no change to intense magenta, in the phloroglucinol and hydrochloric acid test for specimen #2, it is possible that there is a mixture of fibre types in the cord. Varying degrees of processing can also result in varying amounts of lignin present in a sample. Degradation can cause loss of lignin as well, but this specimen was not degraded. The various colour changes caused by the Herzberg reagent, however, are typical of more than one fibre type. cuprammonium hydroxide gave swelling and dissolution patterns for flax, hemp and probably jute. The twist test showed that there were clockwise and counterclockwise rotating fibres and indicated the presence of more than one fibre variety. The fibre diameter measurements were consistent with a mixture of fibres, as well. The mean diameter (48.8 μm) is typical of fibre measurements found in hemp and jute. The large

Table 28
Specimen #1 Napkin from Mrs. H. Bentley, approx. 1900

Original Colour	White
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Reddish brown, intense yellow extraneous material, many different types of nodes deeply stained, no visible lumina
Schweitzer's Reagent	Swelling and dissolution as for flax with remaining protoplasmic ribbon; has S fibrillar orientation
Twist Test	Counterclockwise
Fibre Diameters	Warp (single cells) Mean: 13.3 µm (n=50) Standard deviation: 5.3 µm Weft (single cells) Mean: 9.7 µm (n=50) Standard deviation: 4.6 µm

Table 29
Specimen #2 Cordage from Mrs. H. Bentley

Original Colour	Light Brown, between Pantone® 465 and 466
Phloroglucinol and Hydrochloric Acid	Some general areas have no colour change, others are so intensely magenta that no Pantone® number could be assigned. Under microscope, just a few bundles of fibres are intensely magenta
Herzberg Reagent	Mostly deep violet, a few reddish violet and a few greenish brown; nodes black, lumina difficult to see
Schweitzer's Reagent	Has fibres with swelling and dissolution as for flax and hemp, has a few fibres that dissolve like jute, but very slowly, has S and Z fibrillar orientation
Twist Test	Has both clockwise and counterclockwise rotating fibres
Fibre Diameters	Single fibre cells and bundles Mean: 48.8 µm (n=50) Standard Deviation: 43.9 µm

standard deviation (43.9 μm) indicates some very fine fibres were found as in hemp. In this case, the fine fibres could also be flax. This cordage specimen appears to consist of flax, hemp and jute when compared with the known specimens in this study.

Specimen #3 was reddish brown and very degraded. Colour changes in the phloroglucinol and hydrochloric acid test are hard to evaluate because of the original reddish brown of the specimen. The exception was the intense yellow extraneous material which is most typically found with flax and hemp. Lumina containing air are most often found in jute. In this case, the cells may be degraded from within, thus opening them up and allowing air bubbles. Fibre diameter measurements are not helpful for this specimen. They come closest to jute, though it is not possible to tell if the clusters of fibres are truly bundles. Even though the twist test was counterclockwise, indicating flax or ramie, only one fibre could be found that was not too degraded to be tested. Schweitzer's reagent was not helpful because the reaction was difficult to see.

The argument against jute, or any other species, as an identification for this specimen is the lack of clarity of the results for all of the tests. Although this fibre did not respond well to testing because of extreme degradation, it is most probably flax. The general appearance of this fibre under optical microscopy is most like flax. Jute is an unlikely choice for a sail, given its lack of strength and propensity to degrade in ultraviolet light and moisture.

Specimen #4 contains no lignin because the phloroglucinol and hydrochloric test gave no colour change; however, the fibres are in bundles, thus indicating that they are probably adhering together because of encrustations. Results with Herzberg reagent were somewhat vague, giving the deep violet and reddish brown that are possible for flax and ramie. Schweitzer's reagent gave more definite results, showing the swelling and dissolution pattern for flax. The twist test was positive for flax and ramie. Though there were some encrusted bundles present, the fibre diameter means and standard deviations were closest to flax. Specimen #4 has the greatest probability of being flax when compared with the four known fibre varieties in this study.

Table 30
Specimen #3 Fibres from Sail from Bishop Museum, Honolulu

Original Colour	Reddish brown, darker than Pantone® 146U
Phloroglucinol and Hydrochloric Acid	Extremely dark brown, too dark for Pantone® number Extremely dark brown under microscope
Herzberg Reagent	Yellowish to greenish brown with some reddish brown, intense yellow extraneous material, lumina containing air
Schweitzer's Reagent	Difficult to see reaction; fibrillar orientation not visible because of encrustations; most cells break away from bundles and then dissolve; some heavily encrusted bundles have no reaction except dissolving at ends
Twist Test	Only 1 fibre long enough to test - counterclockwise
Fibre Diameters	Too few fibres to measure warp and weft separately Mean: 48.8 µm (n=50) Standard Deviation: 18.1 µm

Table 31
Specimen #4 Canvas Fabric from Canadian Conservation Institute

Original Colour	Warm grey, approx. Pantone® 36 but with a lot of variation
Phloroglucinol and Hydrochloric Acid	No colour Change No colour observable under microscope
Herzberg Reagent	Mostly deep violet, some reddish brown, some yellow, a few black areas; very broken up; a few nodes of swollen type
Schweitzer's Reagent	Swelling and dissolution as for flax; only a few fibre cells show fibrillar orientation which is S
Twist Test	Counterclockwise
Fibre Diameters	Warp (single cells and bundles) Mean: 15.2 µm (n=50) Standard Deviation: 7.2 µm Weft (single cells and bundles) Mean: 14.8 µm (n=50) Standard Deviation: 7.3 µm
EDXA	Presence of calcium in crystals on surface

There was no apparent colour change when specimen #5 was tested with phloroglucinol and hydrochloric acid, thus no presence of lignin is indicated. It is possible that a small change had taken place and was masked by the reddish brown of the fibres. As well, the burgundy colour present when testing was done with Herzberg reagent could be partly attributed to the fibres' colour. With this taken into account, the colour change indicates flax, hemp or ramie. Schweitzer's reagent gave the results of swelling and dissolution for flax. The twist test was positive for flax and ramie. Fibre diameter means and standard deviations came closest to flax for the warp, and to ramie for the weft. The warp figures are very close to those of flax with the frequency distribution pattern being almost identical in spite of specimen #5 having small bundles as well as single cells. This fibre specimen has a greater possibility of being flax than any other known fibre in this study.

Specimen #6 contains no lignin as there was no colour change when it was tested with phloroglucinol and hydrochloric acid. Because the Herzberg reaction was positive for flax and hemp and this specimen has single cells and twisted counterclockwise, an identification of flax is indicated. This is reinforced by the swelling and dissolution pattern which was the same as that of flax. Fibre diameter means and standard deviations were close to those of flax. This specimen tested consistently positive for flax when compared with the known specimen tests in this study.

Specimen #7 contains no lignin as there was no colour change when it was tested with phloroglucinol and hydrochloric acid. Herzberg reagent produced colour consistent with flax, hemp and ramie. The visible narrow lumina are most consistent with flax. The counterclockwise rotating caused by the twist test indicates flax or ramie, while both testing with Schweitzer's reagent and the fibre diameter means and standard deviations came closest to this specimen testing positive for flax.

Plate 5 (c) is an SEM photograph of specimen #7. It shows the typical bamboo-like swollen nodes of flax and fibrillar orientation in an S direction. Other types of nodes are

Table 32
Specimen #5 Marine Fabric from Canadian Conservation Institute

Original Colour	Pale reddish brown, approx. Pantone® 153U but with darker areas
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Brownish violet, almost burgundy; has nodes of all types, obvious lumina of all widths, some with air bubbles
Schweitzer's Reagent	Swelling and dissolution as for flax with some ballooning and many encrustations; has S fibrillar orientation
Twist Test	Counterclockwise
Fibre Diameters	Warp (single cells and small bundles) Mean: 14.6 µm (n=50) Standard Deviation: 5.3 µm Weft (single cells and small bundles) Mean: 18.1 µm (n=50) Standard Deviation: 7.4 µm

Table 33
Specimen #6 Fabric from Pillow Slip, J. Marshall

Original Colour	White
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Deep purple, black nodes, intense yellow extraneous tissue; nodes of all types, has narrow lumina
Schweitzer's Reagent	Swelling and dissolution as for flax; has S fibrillar orientation
Twist Test	Counterclockwise
Fibre Diameters	Warp (single cells) Mean: 13.9 µm (n=50) Standard Deviation: 5.6 µm Weft (single cells) Mean: 14.4 µm (n=50) Standard Deviation: 7.3 µm

not visible as SEM photographs are topographical and do not show interior structures. This specimen is remarkably clean and shows little degradation considering its great age.

Specimen #8 contains no lignin as no colour change took place when it was tested with phloroglucinol and hydrochloric acid. It is difficult to determine a specific meaning from the reaction with the Herzberg Reagent as there were a variety of colours present including dark violet and dark brown nodes. There was not a corresponding variety of fibres present when judged by microscopic appearance alone. They seemed quite homogeneous. In Schweitzer's reagent, the specimen swelled and dissolved like flax, only more rapidly. This can happen when fibres are somewhat degraded, leaving the cellulose component in a purer form. The twist test was counterclockwise indicating flax or ramie. Fibre diameter means and standard deviations came closest to flax. Of the four known species in this study, this specimen comes closest to being flax.

The results for the tests administered to specimen #9 are essentially the same as those for specimen #8. They are both from the same garment with specimen #9 being washed. The more intensive staining observed with Herzberg reagent could be due to easier accessibility caused by the removal of oxidized cellulose products during washing. The fibres rotated in a counterclockwise direction for the twist test. Fibre diameter means and standard deviations vary slightly from those of specimen #8 but are still closest to those of flax. The fibre diameter mean varies by only 1.4 μm from that of specimen #8.

Specimen #10 is highly lignified as it became magenta when tested with phloroglucinol and hydrochloric acid. Though the colour changes with Herzberg reagent are somewhat ambiguous, they are most like jute of the four known species in this study. Typical, jute-like cell ends and lumina with air bubbles were also highlighted. The twist test gave indefinite results as only one fibre could be found that would rotate. Possibly the high degree of lignification interfered with rotation. It can be seen from the SEM photograph (see Plate 5, a) that a large amount of plant tissue is present. Remnants of adjacent cells, probably parenchyma, still adhere to the fibres. The fibre diameter mean of 58.2 μm and standard deviation of 36.6 μm give indefinite results as well with the mean being closest to hemp and the standard deviation closest to jute. The frequency pattern is closer to that of hemp than that of jute. In cuprammonium hydroxide this fibre

Table 34
Specimen #7 Fibres from Egyptian Fabric, Royal Ontario Museum, 1450 B.C.

Original Colour	Pale yellow beige
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Deep violet; nodes of all types, some surface encrustations but in good condition; has difficult to see narrow lumina
Schweitzer's Reagent	Swelling and dissolution as for flax; has S fibrillar orientation but can see it only in a few cells
Twist Test	Counterclockwise, only 2 samples long enough to perform test
Fibre Diameters	Single fibre cells and small bundles Mean: 15.2 µm (n=50) Standard Deviation: 6.8 µm

Table 35
Specimen #8 Fibres from Child's Coptic Tunic, Royal Ontario Museum (before washing)

Original Colour	Brownish golden yellow, Pantone® 139U
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Pale golden brown to dark brown, some violet, some nodes are dark violet, some are very dark brown; many nodes and narrow lumina (unique stain pattern for this study)
Schweitzer's Reagent	Swelling and dissolution as for flax, but dissolves very quickly; fibrillar orientation is not visible
Twist Test	Counterclockwise
Fibre Diameters	Single cells and small bundles Mean: 13.5 µm (n=50) Standard Deviation: 4.5 µm

Table 36
Specimen #9 Fibres from Child's Coptic Tunic, Royal Ontario Museum (after washing)

Original Colour	Brownish golden yellow, Pantone® 139U
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Same as specimen #8, but more intensely stained
Schweitzer's Reagent	Swelling and dissolution as for flax, same as #8; fibrillar orientation is not visible
Twist Test	counterclockwise
Fibre Diameters	Single cells and small bundles Mean: 12.9 µm (n=50) Standard Deviation: 5.04 µm

Table 37
Specimen #10 Fibres from Royal Ontario Museum

Original Colour	Brown, similar to Pantone® 146U
Phloroglucinol and Hydrochloric Acid	Deep magenta, Pantone® 194U From pale pink to deep magenta under microscopy
Herzberg Reagent	Very opaque but has violet, greenish and yellowish brown areas; extraneous tissue is deep violet; has bundles with pointed ends and air bubbles like jute; can see lumina and nodes with difficulty
Schweitzer's Reagent	Surface material breaks up around fibres, then bundle cells break up into segments which swell but do not dissolve; very broad empty lumina, unlike swelling and dissolution pattern for flax, hemp, jute or ramie
Twist Test	1 fibre - clockwise No others could be found that moved
Fibre Diameters	Bundles Mean: 58.2 µm (n=50) Standard Deviation: 36.3 µm

reacted unlike flax, hemp, jute or ramie. The fibres first broke up into segments which then became swollen, rather than swelling first. They did not completely dissolve. For this reason, no definite identification can be made for this specimen. It is, most probably, a vegetable fibre not investigated in the known specimens of this study.

Specimen #11 does not contain lignin as no colour change occurred when it was tested with phloroglucinol and hydrochloric acid. The reaction to Herzberg reagent was most typical for flax and hemp, becoming deep violet and reddish violet, particularly because of the yellow extraneous material. Ramie specimens did not show this colour and ramie usually has some wider lumina. The twist test was positive for flax and ramie, while Schweitzer's reagent showed the swelling and dissolution pattern consistent with flax. Fibre diameter means and standard deviations were also closest to flax, even though some small bundles were included. This specimen is most probably flax as test results are consistent with those of the known specimens of flax tested in this study.

Specimen #12 contains no lignin as no colour change took place when it was tested with phloroglucinol and hydrochloric acid. Herzberg reagent caused violet and brown colour changes with darker nodes, colour changes consistent with flax and hemp, which both contain yellow extraneous material. Hemp can contain areas of fibres that stain brown as well. The large number of nodes and the narrow lumina are most typical of flax. The counterclockwise rotation of the twist test is consistent with flax and ramie. Schweitzer's reagent caused a reaction consistent with that of flax but without visible fibrillar orientation. Fibre diameter means and standard deviation are of little use because the specimen contains only single cells and large bundles.

The SEM photographs in c of Plate 5 of this specimen show fibres in bundles. There is some debris on the fibre surfaces. The bundle formation seems more consistent with hemp and the nodes appear more like those of ramie as can be seen in b and c of Plate 4. This specimen, while testing as flax in most cases, can not be given a conclusive identification because of the presence of large bundles. Single cells and bundles were tested in all cases. This specimen is possibly a blend of flax and some other vegetable fibre.

Table 38
Specimen #11 Fabric from Conservation Lab, University of Alberta

Original Colour	Pale beige, paler than Pantone® 468U
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Deep violet and reddish violet, some intense yellow extraneous material; all types of nodes, occasional narrow lumina
Schweitzer's Reagent	Swelling and dissolution as for flax; has S fibrillar orientation but can see it in only a few fibres
Twist Test	Counterclockwise
Fibre Diameters	Warp (single cells and small bundles) Mean: 15.4 µm (n=50) Standard Deviation: 5.5 µm Weft (single cells and small bundles) Mean: 12.3 µm (n=50) Standard Deviation: 5.0 µm

Table 39
Specimen #12 Fibres from Royal Ontario Museum

Original Colour	Pale yellowish grey, between Pantone® 4505U and 4515U
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Violet and brown, nodes are dark violet or dark brown, intense yellow extraneous material; all types of nodes, occasional narrow lumina in single cells
Schweitzer's Reagent	Swelling and dissolution as for flax but without visible fibrillar orientation
Twist Test	Counterclockwise
Fibre Diameters	Single cells and large bundles Mean: 21.1 µm (n=50) Standard Deviation: 13.4 µm

Specimen #13 contains no lignin as no colour change occurred when it was tested with phloroglucinol and hydrochloric acid. Results from testing with Herzberg reagent showed deep violet with intense yellow extraneous material which is consistent with flax as were results from testing with Schweitzer's reagent. The twist test was positive for flax and ramie, rotating counterclockwise. The fibre diameter mean of 12.8 μm and standard deviation of 4.7 μm came closest to that of flax. An identification of flax can be given to this specimen when compared with the four species in this study.

Specimen #14 is probably very slightly lignified as it had a few pink areas when tested with phloroglucinol and hydrochloric acid and observed under optical microscopy. This specimen is very encrusted and degraded. The degradation can be seen in Plate 6, (a). The colours produced by Herzberg reagent give indefinite results with violet and brownish violet, except that the reagent highlights very narrow lumina, most typical of flax. Though Schweitzer's reagent causes dissolution as for ramie, a thread of protoplasm remained like that in flax. Most probably, degradation has caused loss of non-cellulosic material, allowing more rapid dissolution of the fibres. The twist test could not be administered because the fibres broke and were to short to test. The fibre diameter mean and standard deviation are not very helpful although even with the presence of encrusted bundles, these measurements are closest to flax with a mean of 11.8 μm and a standard deviation of 3.6 μm. An identification can only be tentatively made with this specimen, as there are a few characteristics that strongly suggest flax.

Specimen #15 contains no lignin as no colour change took place when it was tested with phloroglucinol and hydrochloric acid. The colours caused by Herzberg reagent (violet with a small amount of brownish violet) are consistent with flax, hemp and ramie, but features were highlighted such as narrow lumina and many nodes of various types. These characteristics are most like those found in flax. Schweitzer's reagent caused swelling and dissolution as in flax, but without visible fibrillar orientation. Results of the twist test were counterclockwise which is consistent with flax and ramie. The fibre diameter mean is similar to that of flax at 15.7 μm while the standard deviation is closest to ramie at 11.8 μm. Single cells as well as small and large bundles were measured for these analyses.

Table 40

Specimen #13 Fibres from Torrington Pant Fabric, #84.8, Franklin Expedition, 1846-48

Original Colour	Light brown, Pantone® 466U
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Deep violet, intense yellow extraneous material; narrow lumina, all types of nodes
Schweitzer's Reagent	Swelling and dissolution as for flax; has S fibrillar orientation
Twist Test	Counterclockwise
Fibre Diameters	Single cells and small bundles Mean: 12.8 µm (n=50) Standard Deviation: 4.7 µm

Table 41

Specimen #14 Canvas Fibres, #82.52, Franklin Collection

Original Colour	Mix of colours from off white to pale yellow to dark grey
Phloroglucinol and Hydrochloric Acid	No colour changes Shows a few deep pink fibre bundles with no colour observable in the rest under the microscope
Herzberg Reagent	Violet, some brownish violet, has visible single cells as well as bundles (encrusted together?), surface encrustations; a few visible very narrow lumina
Schweitzer's Reagent	Single cells break up and dissolve without swelling (like ramie), some snake-like threads of protoplasm remain, but many cells show S fibrillar orientation
Twist Test	Too brittle to be tested
Fibre Diameters	Bundles Mean: 11.8 µm (n=50) Standard Deviation: 3.6 µm

Table 42
Specimen #15 Canvas Fibres, #82.3, Franklin Collection

Original Colour	Mix of colours from off white to pale yellow to dark grey
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Violet with small amount of brownish violet, surface encrustations; single cells have a few very narrow lumina and nodes like those in flax
Schweitzer's Reagent	Swelling and dissolution as for flax, but few cells react because of encrustations; fibrillar orientation is not visible
Twist Test	Counterclockwise
Fibre Diameters	Single cells and bundles Mean: 15.7 µm (n=50) Standard Deviation: 11.8 µm
EDXA	Presence of calcium in crystals on surface

Table 43
Specimen #16 Cordage, #82.62, Franklin Collection

Original Colour	Grey, close to Pantone® 454U
Phloroglucinol and hydrochloric Acid	Approx. 70% has no colour change, 30% is light magenta; shows strands of magenta throughout under microscope
Herzberg Reagent	Deep reddish violet with some violet and some brown, much darker nodes; almost all encrusted bundles with cells breaking away, a few nodes visible in cells that have pulled away
Schweitzer's Reagent	Impossible to determine reaction as bundles and single cells are too heavily encrusted
Twist Test	Too brittle to be tested
Fibre Diameters	Too degraded and encrusted to measure fibre diameters

Because of the microscopic appearance and results of the various tests, especially Schweitzer's reagent, this specimen is more likely to be flax than any other fibre in this study.

Specimen #16 has some areas of lignification and some areas without as can be seen by the selective magenta colour changes that took place when testing was done with phloroglucinol and hydrochloric acid. Herzberg reagent gave no definite indication of fibre type, either through the reddish violet, violet and brown colour changes or through highlighting fibre characteristics. When Schweitzer's reagent was applied to the fibres, either no reaction occurred because the solvent could not penetrate the encrustations or the reaction could not be seen because of the encrustations. Neither the twist test nor the measurement of fibre diameters could be done because of the high degree of degradation of this specimen. Plate 6, (b) in the SEM photographs shows the degradation and encrustation of specimen #16. Because so little information can be gathered from testing and observing this specimen, historical sources giving possible origins and uses of this cordage could be useful in helping to identify the fibre species.

Specimen #17 does not contain lignin as no colour change took place when it was tested with phloroglucinol and hydrochloric acid. Testing with Herzberg reagent gave positive results for flax and hemp by turning deep violet with intense yellow extraneous material. Schweitzer's reagent, in spite of heavy encrustations, gave swelling and dissolution patterns like those of flax showing S fibrillar orientation. The twist test was less useful but showed counterclockwise rotation in two specimens. No other fibres could be found that were strong enough to undergo the test. Fibre diameters could not be measured because of extreme encrustation and degradation. From the test results collected, this specimen is more like flax than any other fibre specimen in this study.

Specimen #18 contains no lignin as no colour change took place when it was tested with phloroglucinol and hydrochloric acid. The deep reddish violet colour changes caused by Herzberg reagent are possible for flax, hemp and ramie. This reagent also highlighted some nodes. Swelling in Schweitzer's reagent caused visible ruffling of what appeared to be middle lamellar material like that of hemp and the weak counterclockwise rotation of the fibres are important characteristics in giving this

specimen a tentative identification of hemp. Fibre diameters could not be taken as the specimen was too degraded and encrusted.

Specimen #19 contains no lignin as no colour change occurred when it was tested with phloroglucinol and hydrochloric acid. The only other results available for this specimen are from the reaction with Herzberg reagent. The colour changes of violet with darker nodes and intense yellow extraneous material were typical for flax and hemp. No identification can be given for this specimen. Plate 6, (c) is an SEM photograph showing the degradation, fungal hyphae and large amounts of debris present.

Specimen #20 does not contain lignin as no colour change took place when it was tested with phloroglucinol and hydrochloric acid. The violet and reddish violet colour change in Herzberg reagent possibly indicates flax, hemp or ramie, though brown extraneous material was not present in any of the known specimens. Schweitzer's reagent was more helpful, showing the typical ruffling found in the middle lamella of hemp along with the Z fibrillar orientation of hemp and jute. No other information could be collected. A tentative identification of hemp can be given to this specimen.

Table 46
Specimen #19 Canvas Fibres, #82.7, Franklin Collection

Original Colour	Pale grey, Pantone® warm grey 3U
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Violet, dark nodes, large amount of intense yellow extraneous material; encrusted bundles (encrusted together?) and single cells; nodes of all types and narrow lumina visible
Schweitzer's Reagent	Impossible to determine reaction as bundles and cells are too heavily encrusted
Twist Test	Too brittle to test
Fibre Diameters	Too degraded and encrusted to measure fibre diameters

Table 44
Specimen #17 Cordage, #82.24, Franklin Collection

Original Colour	Pale grey, Pantone® cool grey 2U
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Deep violet with much darker nodes, small amount of intense yellow extraneous material; single cells and encrusted bundles (encrusted together?), narrow lumina and all types of nodes visible
Schweitzer's Reagent	Swelling and dissolution as for flax, except where heavy encrustations occur; has S fibrillar orientation
Twist Test	Counterclockwise, only 2 samples strong enough to be tested
Fibre Diameters	Too degraded and encrusted to measure fibre diameters

Table 45
Specimen #18 Cordage, #82.42, Franklin Collection

Original Colour	Pale grey, Pantone® cool grey 2U
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Very deep reddish violet; a few very encrusted single cells and bundles (encrusted together?), a few nodes visible
Schweitzer's Reagent	Impossible to see most of reaction because of encrustations; fibrillar orientation not visible but ruffling is visible as for middle lamella in hemp
Twist Test	Slightly clockwise, samples are very weak and brittle
Fibre Diameters	Too degraded and encrusted to measure fibre diameters

Table 47
Specimen #20 Cordage, #82.27, Franklin Collection

Original Colour	Pale grey, approx.Pantone® warm grey 3U
Phloroglucinol and Hydrochloric Acid	No colour change No colour observable under microscope
Herzberg Reagent	Violet and reddish violet, nodes much darker, has some light brown extraneous material; encrusted bundles (encrusted together?), nodes of all types and a few narrow lumina
Schweitzer's Reagent	Impossible to see most of reaction because of encrustations but can see some fibrillar orientation which is in Z direction and some ruffling as for middle lamella of hemp
Twist Test	Too brittle to test
Fibre Diameters	Too degraded and encrusted to measure fibre diameters

CHAPTER 5 CONCLUSIONS AND RECOMMENDATIONS

Summary

The most commonly used bast fibres, both now and in the past several thousand years, have been flax, hemp, jute, and ramie. Identity of these fibres is important not only for the textile conservator but for the historian, archaeologist and anthropologist as well, however, the identification of bast fibres is difficult, and often impossible, particularly when they are in degraded states.

Many methods of fibre identification have been developed. They have been used with varying degrees of success, but usually little information about methods is given in the literature. Results of these methods often vary greatly among authors, either because of imprecise technique or because of variation in procedures.

For a test to be successful, it must either highlight unique structural details or cause unique behaviour in a fibre. When fibres are degraded, the detection of any features that persist must be facilitated. Many tests were considered in this research. The tests finally used were selected because of their consistent and precise results when used in trial tests.

This research had three objectives. The first was to develop a scheme that could make possible the conclusive identification of flax, hemp, jute and ramie. If identification could not be definite, a specimen could, at least be placed within a smaller group of fibres. The second objective was to determine whether there were characteristics which persist as flax, hemp, jute and ramie are degraded, and if present, to determine whether the traits were useful in identifying unknown fibres. The third objective of this research was to attempt to determine the identity of twenty unknown hard or soft vegetable fibres using an identification scheme that was developed.

Flax, hemp, jute and ramie specimens from reliable sources served as known specimens. Ten each of flax, jute and ramie and six of hemp were tested and observed. Where specimens were fabric, the warp and weft were tested separately.

General microscopic observation of the known specimens verified that none of them was a blend. Degree of processing and degradation were also observed at this time. Next, these specimens were tested with phloroglucinol and hydrochloric acid for the presence of lignin, Herzberg reagent for lignified tissue and to highlight cell structure,

and Schweitzer's reagent to swell and dissolve cellulose in unique patterns. Colour changes, highlighted fibre structures and fibre behaviour were noted, either under simulated daylight in the Dianolite™ box with the Pantone® colour selector, with the optical microscope or both. Next the twist test was administered to determine fibrillar orientation. Fibre diameter measurements were taken, with statistical analysis including frequencies, means and standard deviations done to establish consistency in these fibre measurements within and between species, if possible. Finally, scanning electron microscopy was employed to observe the fibre specimens. Typical and unique characteristics were noted along with degree of degradation in the unknown specimens.

Generally, the results of the chemical tests and observations of the known fibres were remarkably consistent. Results with phloroglucinol and hydrochloric acid showed that none of the bleached flax fibres contained lignin, and only small amounts were present in the unbleached specimens. The hemp and jute specimens were all lignified, with jute staining more intensely than hemp. Hemp, which is less lignified than jute, stained less and the colour was more uneven than in jute. The ramie contained no traceable lignin as no colour changes took place. These results were consistent with the Textile Institute (1975), Koch (1963) and Harris (1954), though the Textile Institute and Harris sometimes found no colour change in hemp, indicating no presence of lignin. Conversely, these authors sometimes found small colour changes in ramie.

With Herzberg reagent, results were less definite than with phloroglucinol and hydrochloric acid, even though dramatic colour changes took place. Flax and ramie turned deep violet through reddish violet to deep reddish violet. Ramie had some brownish violet. Nodes were highlighted by staining more intensely than the rest of the fibres. This was consistent with most authors, though Koch (1963) and Heyn (1954) reported bluish violet as well. Most important was the increased visibility of lumina which were generally narrower in flax and usually wider and more varied in ramie. The colour developed in hemp varied from violet black to reddish violet or pale pink violet. Lumina were not highlighted, but the many cross-markings on hemp fibres became visible. Both flax and hemp had intensely yellow-stained extraneous tissue. Jute changed

colour uniquely and consistently of the four species, turning greenish brown with some reddish brown. The lumina and cell ends became more distinct.

Schweitzer's reagent caused flax, hemp and jute to swell and dissolve according to their own patterns. Ramie simply dissolved without visible swelling in less than 30 seconds. Flax shrank longitudinally while it expanded laterally causing its S fibrillar orientation to be visible and leaving behind a characteristic thread of protoplasm from the lumen after dissolution. The process was rapid, but slower than ramie. Hemp, which took longer than flax to react, showed a different pattern. First, individual cells with attached middle lamellar material peeled away from bundles after which characteristic ruffling of the middle lamella took place. The direction of fibrillar orientation which was Z was not as easy to see as that of flax. There was no protoplasmic residue after dissolution. The swelling and dissolution process was slower for jute than for any of the other three fibre species. As fibre cells pulled away from bundles, they twisted in the Z direction of fibrillar orientation, taking a strip of middle lamella with them which wrapped around the swelling cell as it twisted. Almost no ramie fibres appeared to undergo swelling and usually each fibre cell dissolved within thirty seconds. No fibrillar orientation could be seen and no protoplasm remained after dissolution.

The twist test gave conclusive results for all four fibre species. Flax and ramie rotated counterclockwise while hemp and jute rotated clockwise. According to Hock (1942), flax and ramie contain fibrils oriented in an S direction with the outermost layer being in a Z direction. Twist is controlled by the underlying fibrils. With hemp and jute, the underlying fibrils are parallel to the fibre axis, while the outer layer of fibrils is oriented in a Z direction. In this case, twist is controlled by the outer layer of fibrils.

Fibre diameter measurements were taken of individual fibre cells for flax and ramie and of cell bundles for hemp and jute because this is the state in which they are most commonly found in yarns and fabrics. Flax was found to have the smallest mean diameter followed by ramie, jute and then hemp. Flax also had the smallest standard deviation indicating that flax fibre diameters are the most uniform of the four. Hemp had the greatest standard deviation of the four species being greater than its mean. It was the least uniform in fibre diameter of the four species.

The group of tests in the identification scheme administered to the known specimens were then administered to the twenty unknown specimens. Results followed a consistent pattern. The less degraded a specimen was, the more conclusive was the identification that could be made. In some cases, however, test results either did not follow those of any of the known specimens or the results for each test and observation of one fibre specimen showed little consistency. Thirteen of the unknown specimens were flax, although some identifications were quite tentative. Five could be given no identification, three were hemp and one was jute. None of the unknowns was identified as ramie.

Conclusions

The possibility of identifying flax, hemp, jute and ramie in textile artifacts was investigated in this research. There is great similarity among bast fibres generally. When they are removed from the surrounding tissue of the plant stem in which they are formed, they can be extremely difficult to identify.

There was consistency of results of the tests done and observations made of the known specimens. It can be seen that careful control of the test procedures using a controlled light source and a colour standard such as the Pantone® colour selector when necessary and experience in conducting the tests results in consistency for this scheme of testing procedures on known specimens.

Several of the unknown specimens were degraded. The more degraded specimens gave less information when tested and observed. Even though not all of the tests were effective on them, the scheme developed should, nonetheless, be administered in the same way for both degraded and undegraded specimens. For instance, some fibres did not respond to the twist test because of degradation and encrustation, where other degraded specimens did. Often lumina and nodes could still be seen in spite of degradation. Unknown specimen #7 clearly showed the bamboo-like nodes of flax, in spite of being 3500 years old. Unknown specimen #12 had visible nodes even when there was a large amount of encrusted debris on the surface of the fibres. Occasionally, darkening of the fibres caused either by oxidation products or possibly dyes, obscured colour changes. One consistent feature of the highly degraded specimens was the encrustation of debris

on the fibres sometimes making it impossible to measure the size of fibre bundles, or to determine whether the fibres were originally in bundles. It is not possible to perceive a progression of breakdown of fibre characteristics as fibres become more degraded. There is no one consistent characteristic that can be depended upon to persist when others are gone though, remarkably, some cell structures or behaviour characteristics could be observed in every unknown specimen in this study. The most consistent of these was nodes.

It was possible to identify fifteen of the twenty unknown specimens with varying degrees of certainty which depended upon consistency of results and amount of information obtainable from the very degraded specimens. Vegetable fibres such as those used in Tapa cloth and for cords and fabrics from various parts of the world need investigation under a scheme such as the one developed in this research. Under such circumstances, it is helpful to know the provenance of the artifact being examined. Most ramie is from China, while most early Egyptian and Coptic vegetable fibres were either cotton or flax.

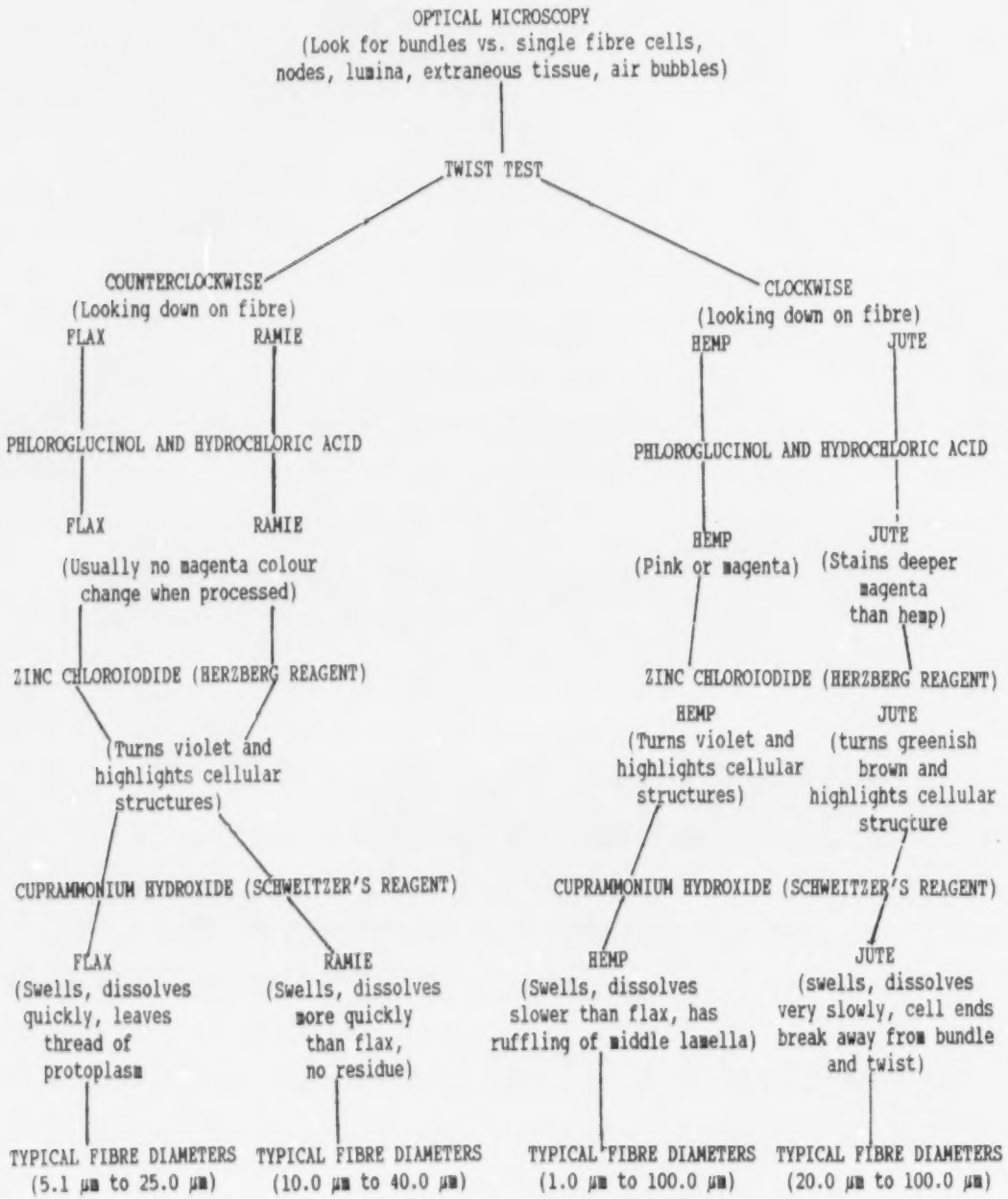
The importance of experience when observing vegetable fibres with the microscope can not be underestimated in observing unknown specimens as the eye becomes trained. If the microscopist has had the opportunity to examine many known specimens of flax, hemp, jute and ramie and has a critical eye for detail, subtle differences can be detected. Such elusive things as the placement of nodes in a bamboo-like pattern on flax or the subtle cross-markings on hemp become easier to detect.

All of the tests and observations proved useful. It is difficult to priorize their usefulness, for their greatest success was in combination with each other. The twist test, was one of the most helpful even though it was not always successful with the more degraded specimens. It gives clear results, needs only simple equipment and is easy to administer. The phloroglucinol and hydrochloric acid test was precise in indicating the presence of lignin, but interpreting that information in terms of species identification was not clear cut, particularly with degraded specimens where originally lignified specimens can sometimes be without lignin. Lignin is more vulnerable to certain types of degradation, particularly that caused by some microbes.

The research undertaken in this project was very successful. The protocol developed can be reliably applied to undegraded bast fibres with the expectation of making a conclusive identification if those fibres are flax, hemp, jute or ramie. If the fibres are slightly degraded, conclusive identifications are usually still possible. Highly degraded fibres are more difficult to identify, but some characteristics can usually be discerned. This information, along with provenance, often allows for conclusive identification.

The following chart is intended as a guide for the administration of the protocol followed in this research. It can be used to identify specimens of flax, hemp, jute and ramie. Known specimens of other hard and soft fibres can also be observed and tested using this scheme, ultimately allowing the identification of more varieties of unknown specimens. Methods for preparation of chemical test reagents can be found in Appendix A-1.

TEST PROTOCOL AND SUMMARY OF OBSERVATIONS



Recommendations for Future Research

The tests administered to the known flax, hemp, jute and ramie specimens were done under well defined, precise conditions. Results were consistent for the known fibres. It would be useful if other known soft and hard vegetable fibres such as sisal and coir were taken through the same testing and observation steps. Dyed fibres could also be administered this scheme. In this way, a broader standard could be developed for comparing the test results of unknown fibre specimens. The resulting information, in combination with the provenance of the unknown specimens would permit a more reliable identification.

The analysis of crystals present in vegetable fibre cells can be done by first ashing the fibres. Either no crystals can be expected to be found, such as in flax or crystals characteristic of various fibres can be expected (Catling and Grayson, 1982). The ashing test can be added to the protocol established in this research.

It is important to note that any further testing of vegetable fibres using this protocol must be done precisely following the methodology described in this project. To be of use results need to be recorded using the same procedures and format along with any additional refinements in technique. Means of cleaning fibres with debris or encrustations such as ultrasonic cleaning done in water or non-polar solvents should be investigated.

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APPENDIX A-1: Chemical Test Preparation Methods

Phloroglucinol and Hydrochloric Acid

Phloroglucinol ($C_6H_3(OH)_3.2H_2O$) and hydrochloric acid (HCl) reagent was prepared according to Harris (1954). A 2% solution of phloroglucinol and 90.5% reagent grade ethanol (CH_3CH_2OH) was made by dissolving 2 g of phloroglucinol in 98 ml of ethanol. This solution was mixed with 100 ml of concentrated hydrochloric acid (HCl).

Herzberg Reagent (zinc chloroiodide)

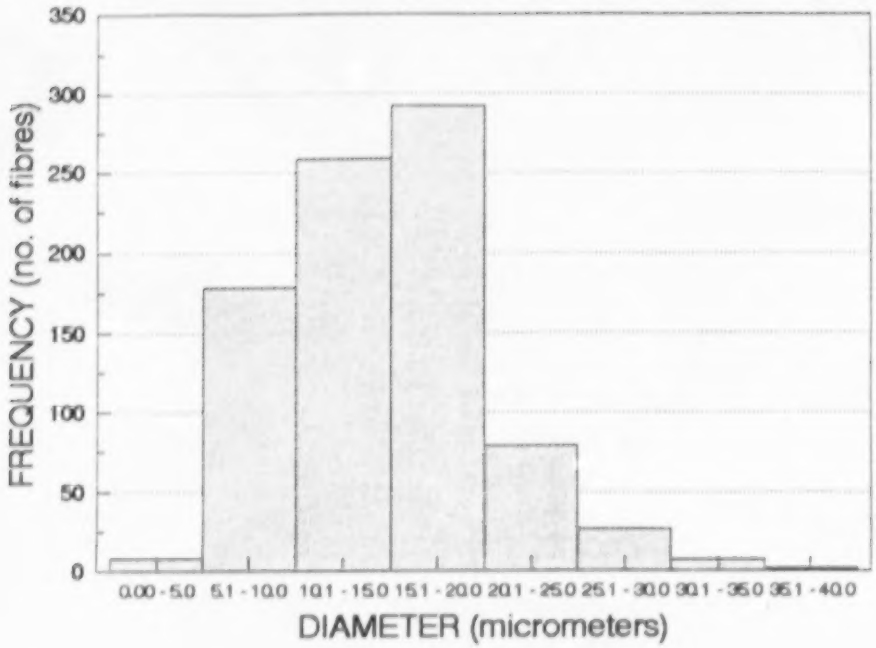
Herzberg reagent was prepared according to the Textile Institute (1975). A solution was made by dissolving 100 g zinc chloride ($ZnCl_2$) in 50 ml water. To this was added 2.1 g potassium iodide (KI) and 5.0 g iodine (I) dissolved in 5 ml of water. A precipitate was formed. After the precipitate had settled the clear liquid was decanted. A leaf of iodine was added to this liquid and it was stored in a dark bottle. The shelf life of this reagent was approximately one week.

Schweitzer's Reagent (cuprammonium hydroxide)

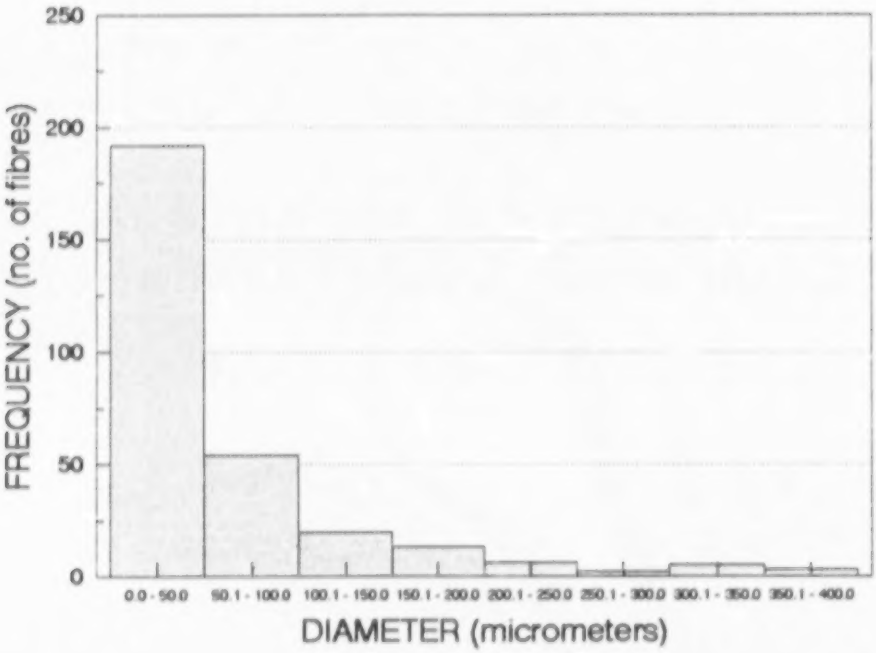
Schweitzer's reagent was prepared according to Koch (1963). A 5% solution of copper sulphate ($CuSO_4$) was prepared by dissolving 5 g of copper sulphate in 100 ml of boiling water. It was cooled. Sodium hydroxide (10% NaOH) was added until all the cupric hydroxide $Cu(OH)_2$ was precipitated out. The precipitate was allowed to settle and the clear liquid was decanted. Distilled water was added with stirring and decanted. The precipitate was filtered and washed thoroughly with distilled water while in the filter paper. It was dried and then powdered finely in a mortar and pestle. A small quantity of the precipitate was dissolved in concentrated ammonium hydroxide (NH_4OH) [eg. 2 g in 100ml of 25% ammonium hydroxide (NH_4OH)] and shaken vigorously.

APPENDIX A-2: Fibre Diameter Frequencies

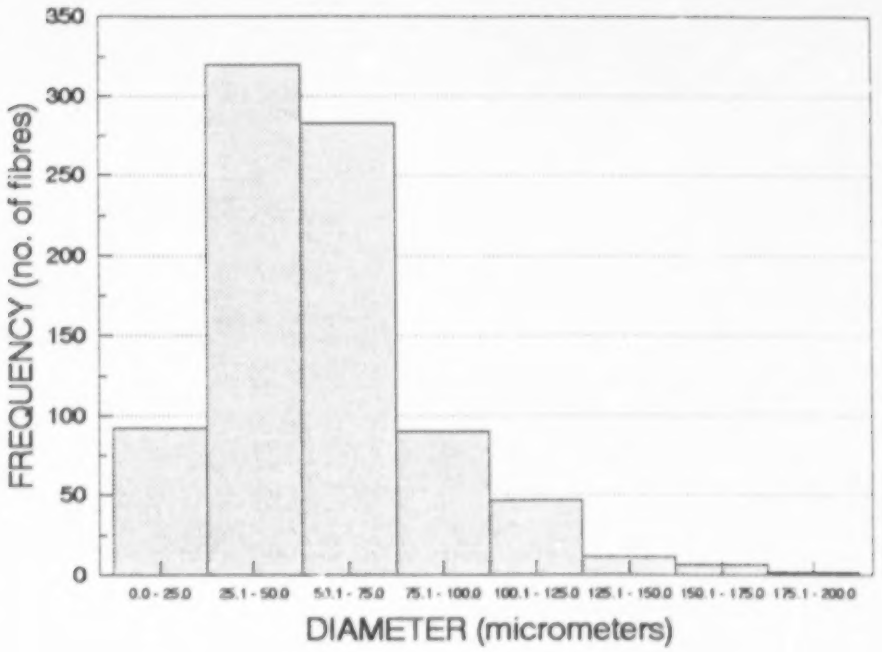
Flax Fibre Diameter Frequencies



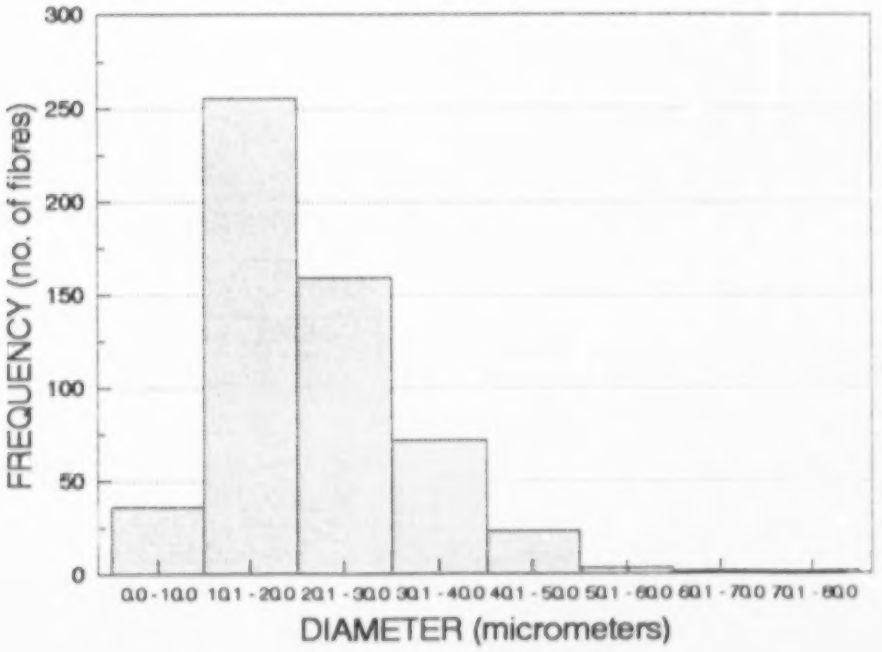
Hemp Fibre Diameter Frequencies



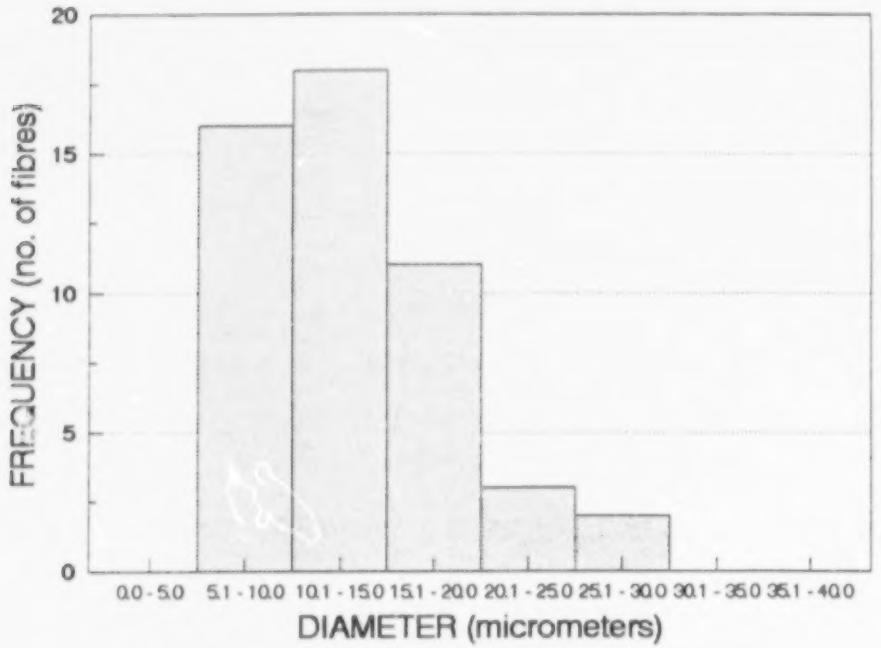
Jute Fibre Diameter Frequencies



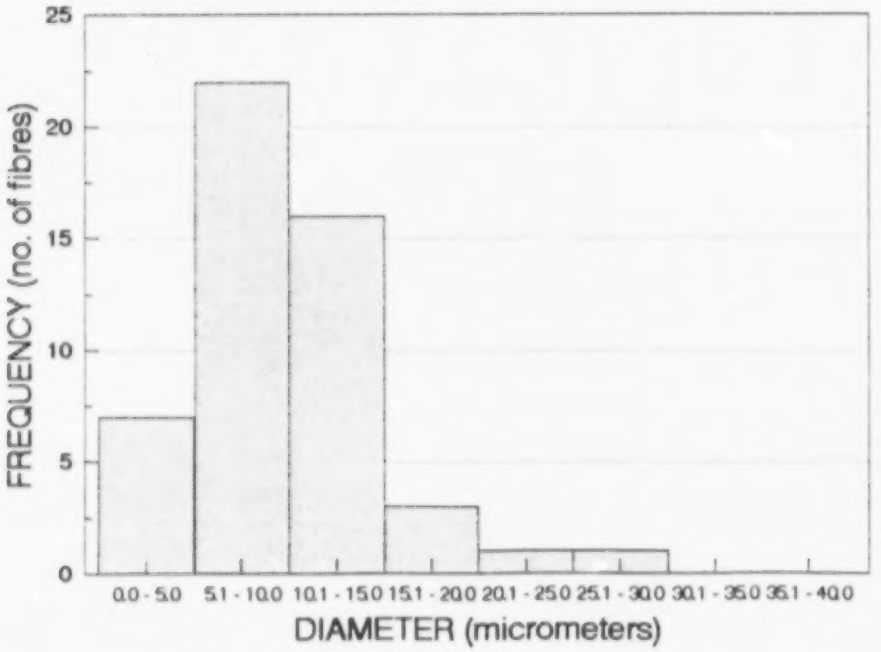
Ramie Fibre Diameter Frequencies



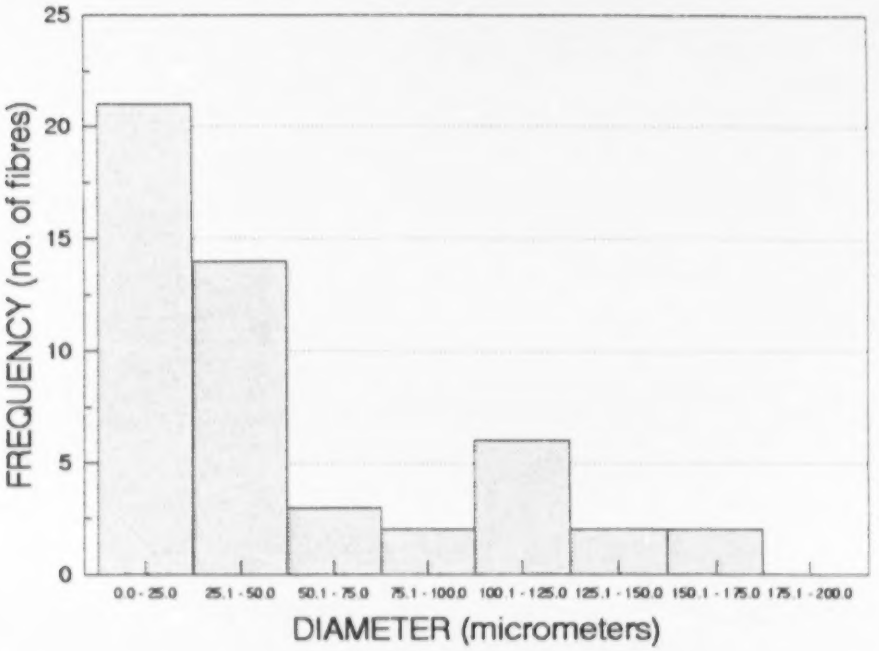
#1 Warp Fibre Diameter Frequencies - Napkin from Mrs. H. Bentley, approx. 1900



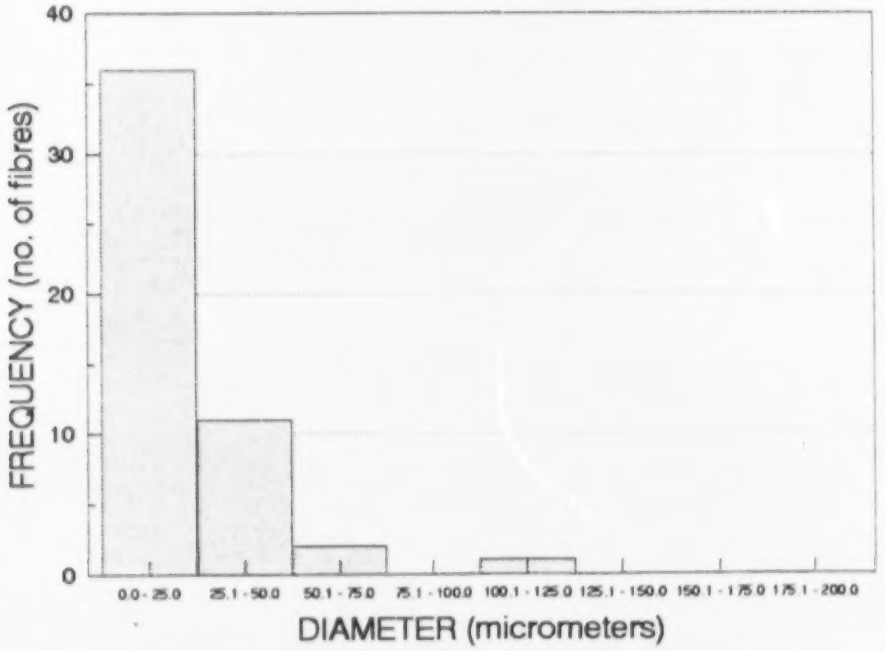
#1 Weft Fibre Diameter Frequencies - Napkin from Mrs. H. Bentley, approx. 1900



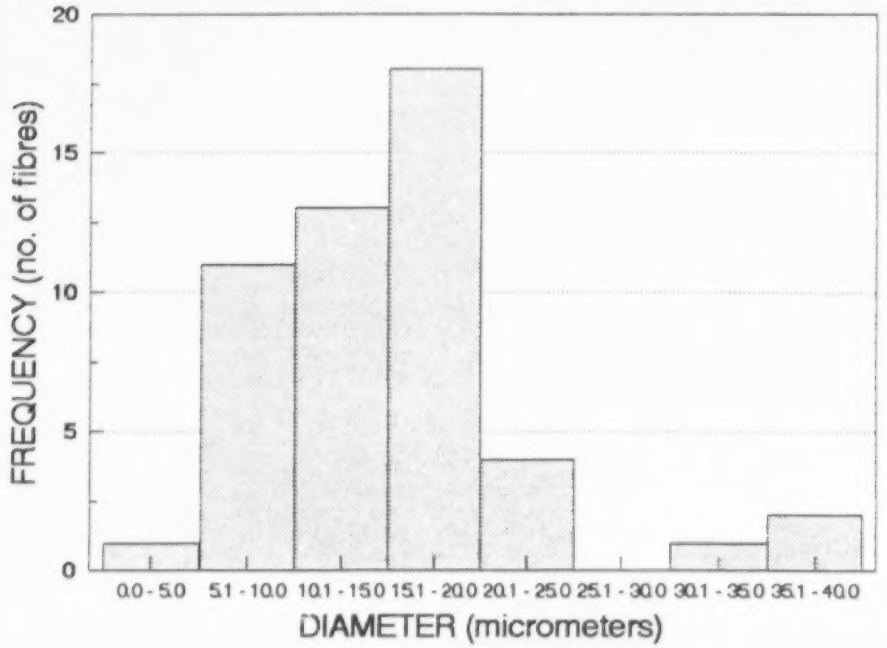
#2 Fibre Diameter Frequencies - Unknown Cordage from Mrs. H. Bentley



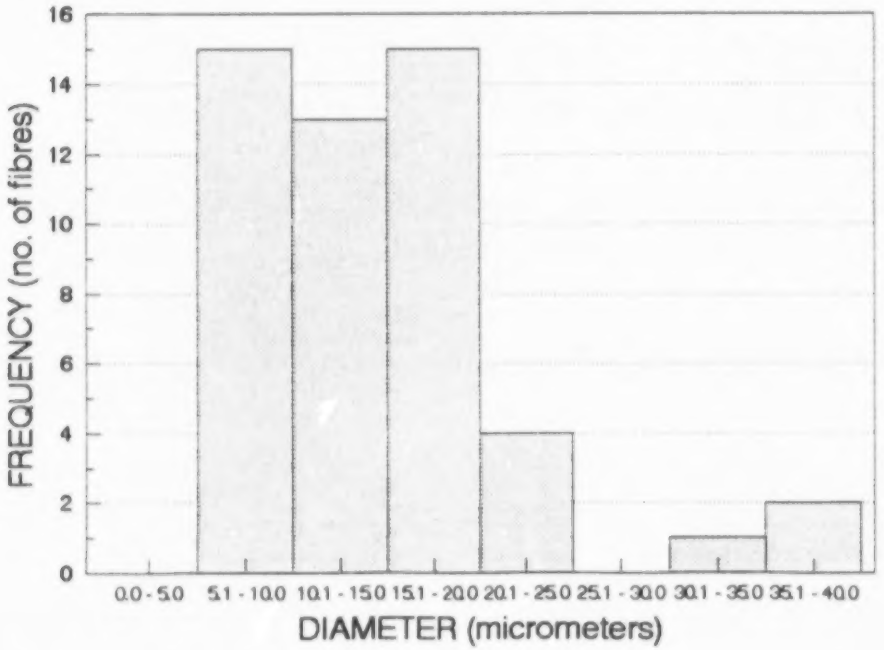
#3 Fibre Diameter Frequencies - Sail from Bishop Museum, Honolulu



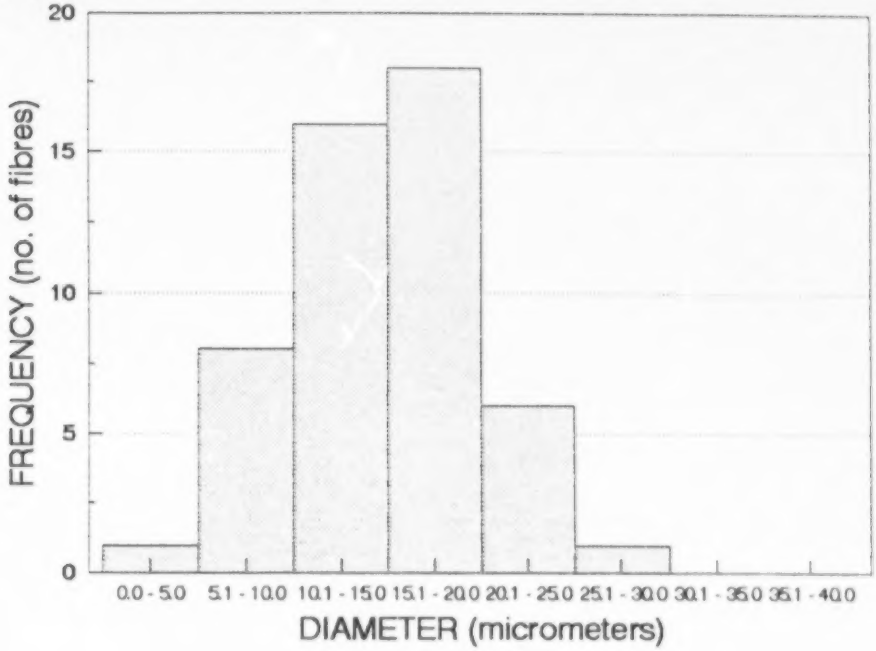
#4 Warp Fibre Diameter Frequencies - Canvas from Canadian Conservation Institute



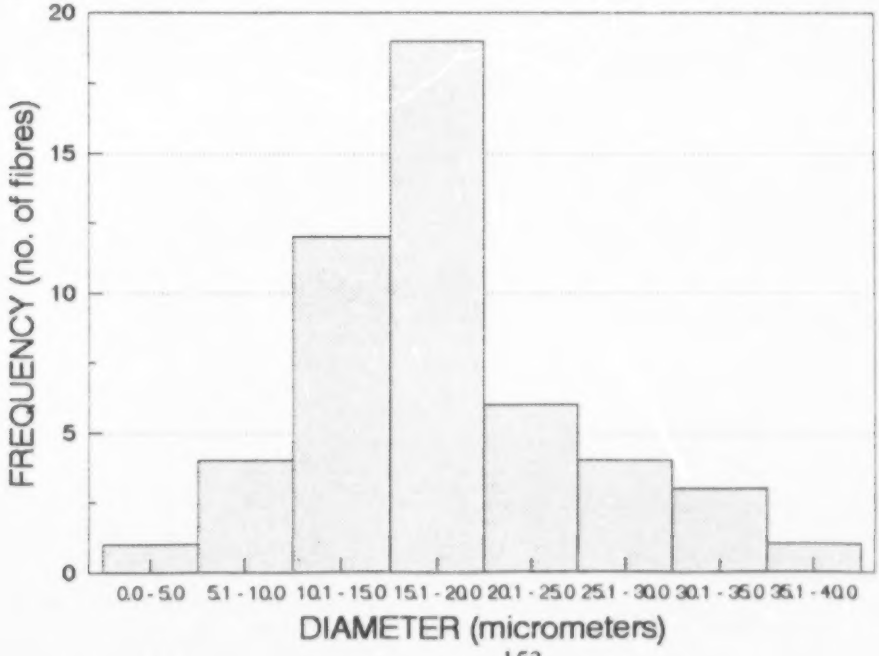
#4 Weft Fibre Diameter Frequencies - Canvas from Canadian Conservation Institute



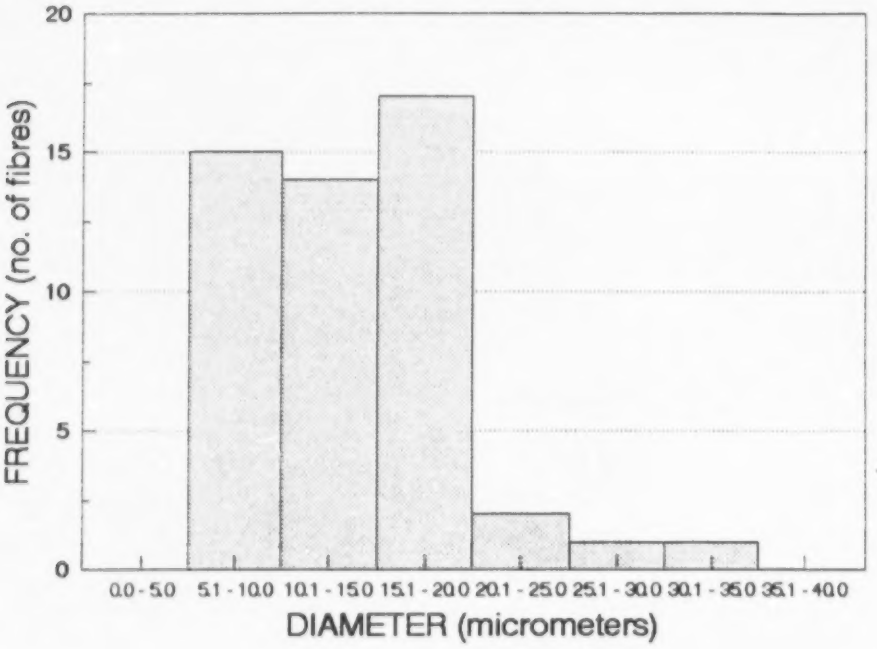
#5 Warp Fibre Diameter Frequencies - Marine Fibres from Canadian Conservation Institute



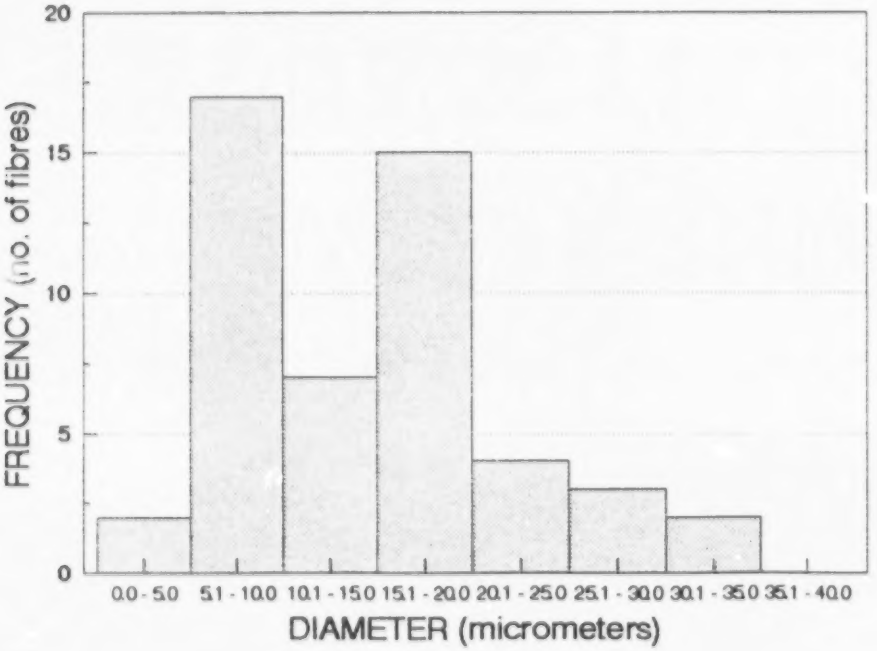
#5 Weft Fibre Diameter Frequencies - Marine Fibres from Canadian Conservation Institute



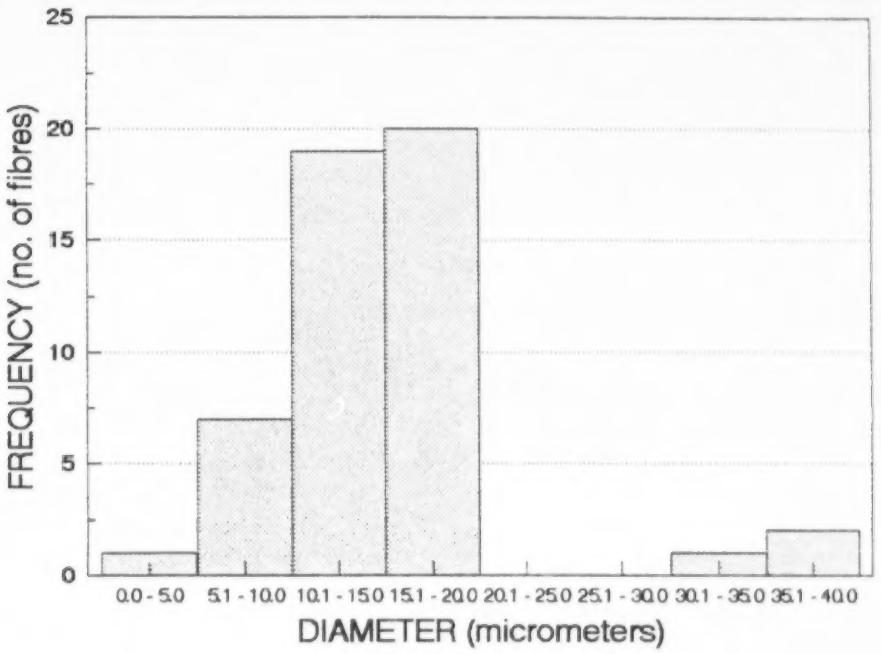
#6 Warp Fibre Diameter Frequencies - Pillow Slip Fabric, J. Marshall



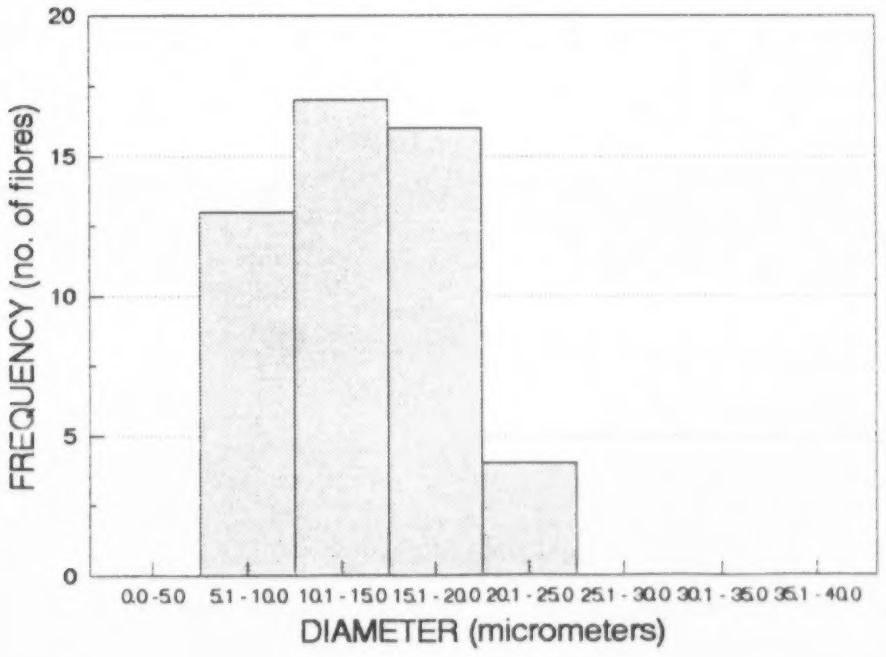
#6 Weft Fibre Diameter Frequencies - Pillow Slip Fabric, J. Marshall



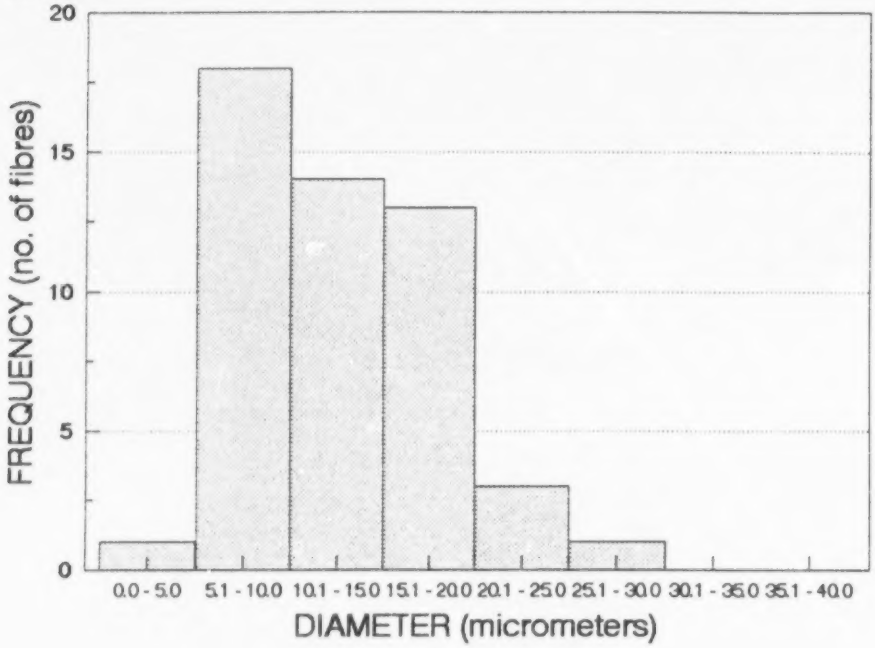
#7 Fibre Diameter Frequencies - Egyptian Fabric, 1450 B.C., Royal Ontario Museum



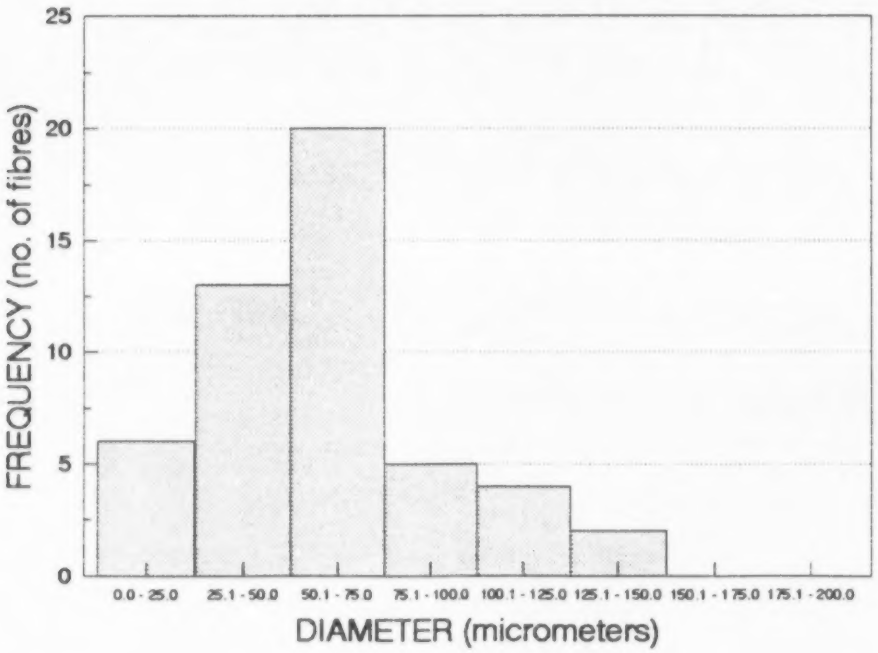
#8 Fibre Diameter Frequencies - Child's Coptic Tunic, Royal Ontario Museum



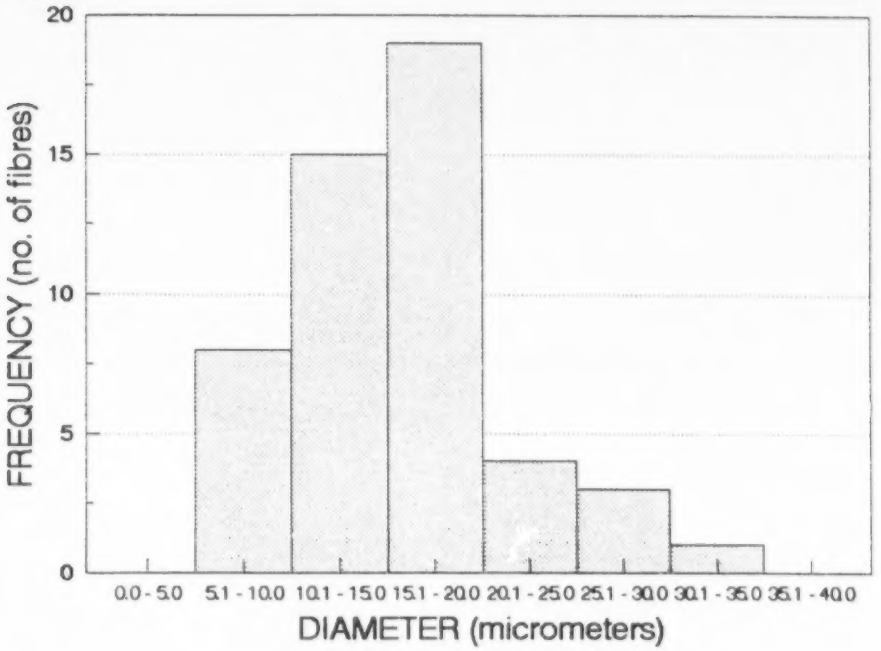
#9 Fibre Diameter Frequencies - Child's Coptic Tunic (after washing), Royal Ontario Museum



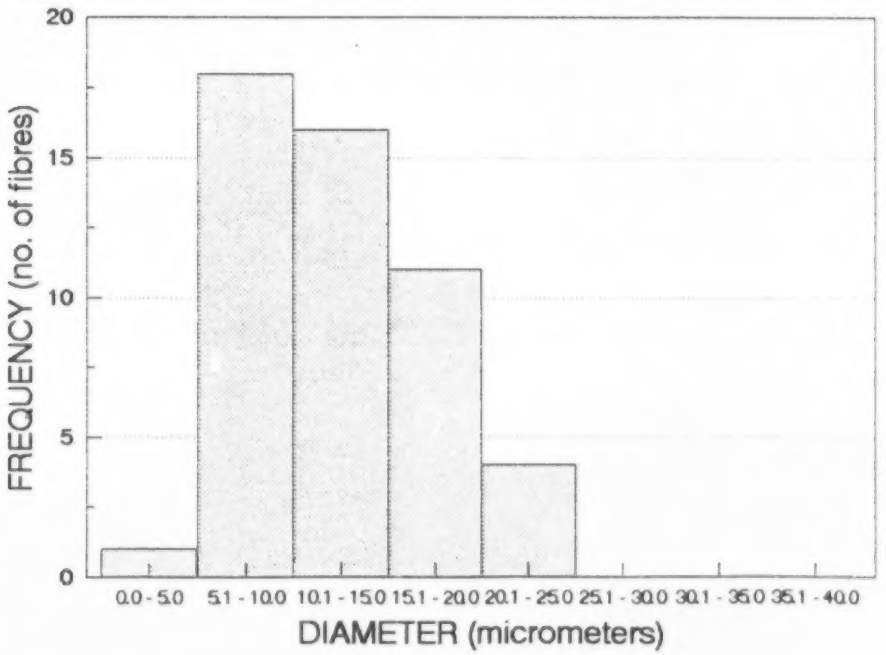
#10 Fibre Diameter Frequencies - Fabric, Royal Ontario Museum



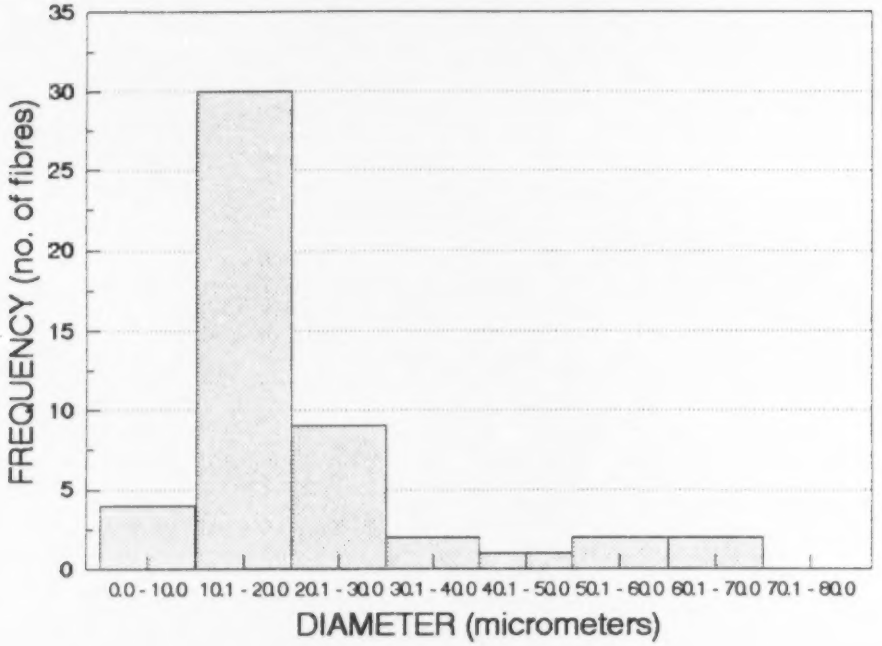
#11 Warp Fibre Diameter Frequencies - Fabric, Conservation Lab, University of Alberta



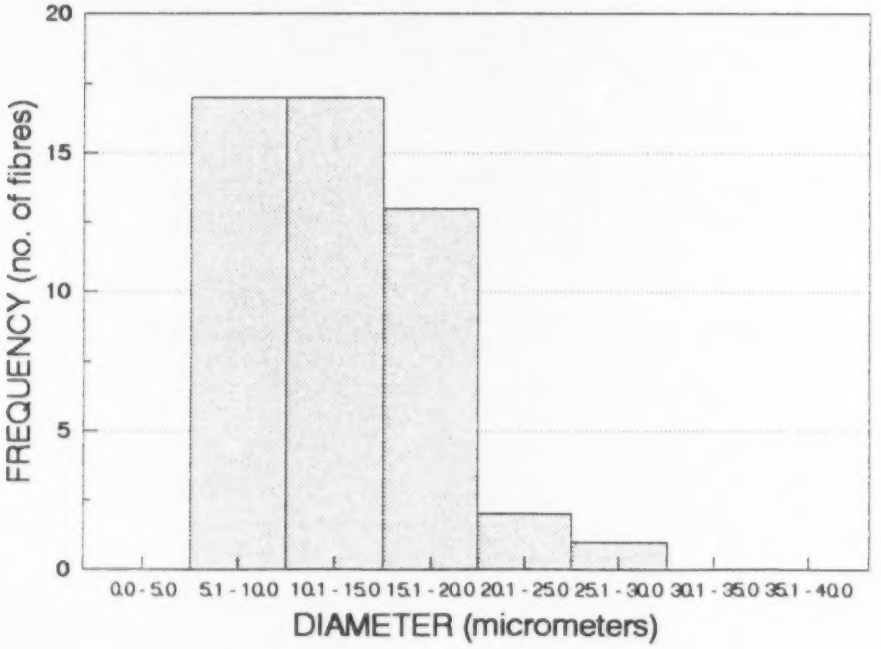
#11 Weft Fibre Diameter Frequencies - Fabric, Conservation Lab, University of Alberta



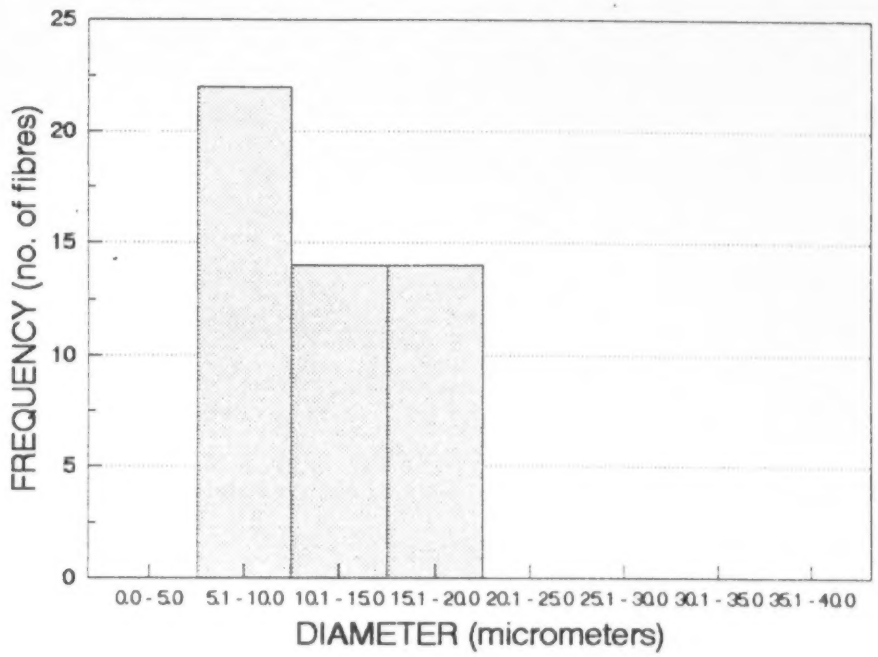
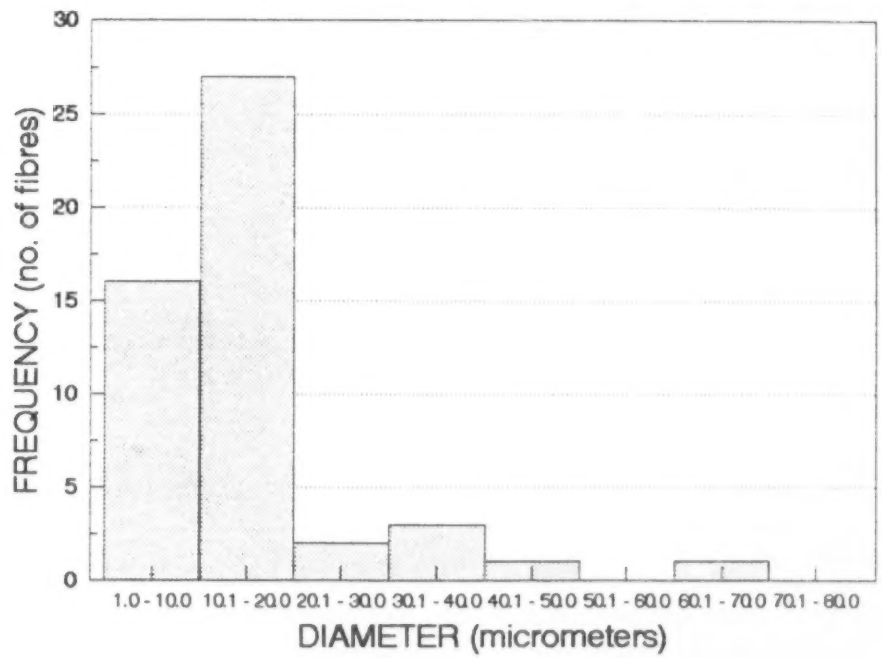
#12 Fibre Diameter Frequencies - Fabric, Royal Ontario Museum



#13 Fibre Diameter Frequencies - Torrington Pant Fabric, Franklin Expedition, 1846



#14 Fibre Diameter Frequencies - Canvas, Franklin Collection



#15 Fibre Diameter Frequencies - Canvas, Franklin Collection